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# Expansion of University Education

**T**HE appointment of a University Commission consisting of eminent educationists from India and abroad "to inquire into, and report on, the conditions and prospects of university education and advanced research in India, and to recommend a constructive policy in relation to the problems they present and the needs of the country", meets an important national need. It is more than three decades ago that a University Commission (The Sadler Commission, 1917) held an inquiry into university and higher education in India, and the vast changes that have taken place since then adds urgency to the detailed examination of the whole field of higher education and research in the country. The preamble to the Government resolution on the appointment of the Commission stresses the need in the following words:

"The importance of primary and secondary education in a democracy is obvious. Their quality, however, depends in the ultimate analysis on the quality of educational leaders, and these can be produced only by a suitable system of higher and university education. Provision of extended facilities for higher education is, therefore, necessary for the satisfactory functioning of a democracy. A vast change has taken place in the country with the achievement of independence. New vistas have opened which necessitate a survey of education at all stages. The interests of the country require vast programmes of expansion in agriculture, industry and commerce, which, however, can be carried out only if a sufficient number of highly trained executives and artisans, craftsmen and skilled labour of all grades and types are available. The problems of defence cannot be satisfactorily solved without the existence, within the country, of all facilities for technological

and scientific studies of the highest order. Extension of the boundaries of knowledge and research is also essential if we are to improve the quality of education which is now available to us."

The 16-page questionnaire issued by the Commission covers 26 different aspects of education and seeks information on such topics as: structure, jurisdiction and administration; admission to courses; medium of instruction; conditions of service of teachers; libraries, laboratories and museums; extra-curricular activities; health and physical education; finances; inter-university relations and attitude to organizations like the U.N.O. and the UNESCO. The composition of the Commission presided over by the distinguished Indian educationist and philosopher, Dr. S. Radhakrishnan, compels confidence, and the very thorough inquiry initiated by the Commission under its comprehensive terms of reference may be expected to lead to results of lasting benefit to higher education in India.

## **Inquiry Should Precede Expansion**

There has been lately a persistent and unanimous demand for the expansion of university education in India. It has been pointed out that the total demand for graduates called for by the Scientific Manpower Committee, and those required by the expanding administrative and social services, cannot be adequately met by the present output of graduates from universities, and that an expansion in the facilities for teaching and research is essential. A quantitative assessment of the demand for graduates is not available, and this renders difficult the determination of the extent and direction of expansion. The real obstacles to any expansion, however, are the material and manpower shortages, and these shortages preclude any great increase in the output of

graduates, at any rate, in the immediate future. The problem of expansion, moreover, needs a good deal of critical thinking as regards both the purpose and content of university education, and the pause necessitated by the material shortages need not be a serious disadvantage, if the opportunity is utilized for investigation, experiment and discussion of the issues involved. The problem should be examined with vision without which "education may settle down to uncreative mediocrity". The temptation to resort to *ad hoc* expedients to increase the number of graduates will have to be resisted in favour of a properly conceived plan of expansion deliberately formulated as a result of fundamental and creative thinking.

#### **Fundamental Objectives Must Be Fostered**

The recognition of the important rôle, which science plays in the promotion of national welfare, has led to a scrutiny of the extent of attention accorded to science in university education. This is to be welcomed. But the view, which seems to be widely prevalent, that universities should be expanded to enable them to provide the trained personnel required for putting into operation "the vast programmes in agriculture; industry and commerce" needs critical examination. This view will have to be considered in relation to such questions as the task of universities in technology, balance of subjects in the faculties, basis for the selection of students and academic standards, and the all-important question of the freedom of universities to formulate their policies and programmes in accordance with their fundamental aims and objects.

"The modern doctrine that the primary function of a university is the supply of experts in the spheres of science, business, administration and social services, can become both dangerous and heretical—", writes Dr. J. Mackay-Mure in *Nature* (1947, March 22, p. 392), "not because it advocates any single thing which ought not to be done, but because it represents an idea of a university which is out of focus. It pushes into the background that conception of it as a centre of cultural life and cultural progress, which makes freedom not merely desirable but a first essential. Without freedom such a centre ceases to be a university, a community of scholars within which every man may open his mind to a total engagement in the struggle for truth... The maintenance of this freedom has today acquired

a new significance. As the State system of education is reformed and developed, there is the assurance of that technical and vocational training on which the efficiency of the community depends. But the quality of the community depends primarily on its standards and its ideals, on the defence and promotion of fundamental values in life and progress. Although standards and values are by no means the monopoly of those who are brought into association with the life of a university, it is doubtful whether they can be maintained if the true idea of a university is allowed to decay." "It is not", says Plato, "the life of knowledge, not even if it includes all the sciences, that creates happiness and well-being, but a single branch of knowledge—the science of good and evil... Science, economics and sociology provide the frames of contemporary society and satisfy its material needs, but unless we have the knowledge of good and evil, their use and excellence will be found to have failed us."

It is our conviction that once the conditions propitious to the furtherance of the fundamental objectives of university education are provided and secured, the universities would be enabled to exert their beneficial influence on society and contribute their due share to the progress of the nation and to the elevation of contemporary civilization. The first responsibility of a university, not only for its own sake, but for the sake of the community, is to *be* a university. It should, in Miss D. M. Emmett's words, be a place where criticism and evaluation of ideas is continually carried forward, where "nonsense" can be exposed for what it is, and where "intellectual virtues" of sincerity of mind are being fostered and transmitted. It should inculcate a sense of values and the power to distinguish what is first rate from what is not. If this sense of values is inculcated and propagated, a proper solution for the many complex problems confronting society would become possible.

#### **Universities & the State**

The rôle of the universities is primarily to impart a liberal education and to instil high ideals of citizenship in the minds of the members of the younger generation and to endow them with the ability to devote their mental and physical powers to constructive ends. University education is a preparation for life and not merely for employment. On this view, the training of experts required for agriculture, industry and commerce

is not the *primary* responsibility of the universities. The State schemes for the development of technological and vocational institutions provide the means for securing "the trained executives and artisans, craftsmen and skilled labour of all grades and types". The Government has the right and the duty to satisfy itself that every field of study which is in the national interest is cultivated in the university system, also that the resources placed at the disposal of universities are properly and efficiently utilized. The Government has the right also to satisfy itself that the universities are under the charge of men who can think imaginatively and creatively and who can be trusted to uphold and carry forward the ideals of higher education. Beyond these, the universities ought to be given the freedom to devise and execute policies in pursuance of their accepted aims and objects.

The universities in India are of diverse types and patterns. Their origins and ages are also diverse. The problems that confront them in their day-to-day working and those that influence their future may be expected to be also diverse. Judging by the questionnaire issued by the Commission, all the aspects which affect the efficiency and progress of the universities are being subjected to scrutiny and inquiry. The conditions now prevailing in India are extremely propitious for the growth and development of university education. There is a

vast reservoir of intelligence in the country which is awaiting to be tapped and which would enable the setting up of high academic standards, provided the crippling disabilities such as lack of means and, in some instances, even caste, which hamper the entry of intelligent youths who have proved their ability to profit by university education, are removed. The constitution of free India provides equal opportunities for all irrespective of caste, creed or sex. A favourable atmosphere for the propagation of the highest ideals of liberal education obtains in India today, thanks to the exalted position which truth and peaceful ideals have attained, largely as a result of Mahatma Gandhi's all-pervading influence. The universities themselves are fully alive to their responsibilities and have shown a readiness to discuss standards of intellectual discipline and their maintenance and propagation. These are healthy signs. In the important task of assessing the present position of university education in India and in determining its future development, the Commission, we feel confident, will have the full co-operation of university teachers, research scholars, administrators and, in fact, of all those who have the vision to see what is at stake and realize how much depends on the universities in providing the nation not only with educated intellectuals but also of men and women of personality and character.

## The Indian Chemical Society— Twenty-five Years' Service to Chemistry

**T**HE *Indian Chemical Society* was established in 1924 as a result of the deliberations held during the annual sessions of the Indian Science Congress, due chiefly to the initiative and foresight of the late Sir P. C. Ray, doyen of chemistry and leader of the Indian school of chemists. He was enthusiastically supported in this venture by the late Dr. E. R. Watson, Dr. J. N. Mukherjee, Dr. J. C. Ghosh, Sir S. S. Bhatnagar, and other leading chemists of the country. The *Society* was registered on the

9th of May 1924, with Sir P. C. Ray as the first President, Dr. G. J. Fowler and Dr. J. L. Simonsen as Vice-Presidents, (the late) Dr. E. R. Watson and Dr. N. R. Dhar as Hon. Editors, Dr. J. N. Mukherjee as Hon. Secretary and Dr. P. C. Mitter as Hon. Treasurer.

The object of the *Society*, broadly speaking, is to cultivate and promote the cause of chemical science and allied branches of learning by holding meetings to discuss papers of scientific interest, arranging lectures on scientific topics, co-operating with other

organizations having similar objects, and publishing original memoirs in chemistry and related branches of science through the medium of the *Society's* publications. These objects the *Society* has fostered zealously, and the status which the science of chemistry has attained in India is in no small measure due to its efforts.

The administration of the *Society* vests in a *Council* consisting of the President, the Vice-Presidents, the Hon. Secretary, the Hon. Treasurer, the Hon. Editors and twenty ordinary members of the *Council* elected by votes from among the Fellows on a regional basis. There are at present nearly 400 Fellows on the rolls. Since 1938 a new class of membership, the Associate membership, was instituted to admit research scholars and post-graduate students. Its membership has been characterized by a continuously upward trend and it enjoys today the support of chemists engaged in every branch of this science and has attained the status and prestige of the premier national organization for the promotion, propagation and cultivation of chemistry in this country.

The *Journal of the Indian Chemical Society*, which was started as a quarterly at its inception, became a bi-monthly in 1927. Since 1934 it is being published every month. Since 1938 the *Society* has been publishing every quarter the *Industrial & News*

*Edition of the Journal*. The adjudication of the publishable matter is entrusted to a Board of Associate Editors consisting of sixteen members including the two Hon. Editors.

The office of the *Society* at Calcutta has attached to it a comprehensive reference library consisting of journals of chemical and allied branches of science and dissertations on scientific subjects. The *Society* at present receives more than 180 scientific publications in exchange, and the holdings of the library include some 2,000 bound volumes of journals dealing with chemical and allied sciences, and 1,200 dissertations. The *Society* supplies to its Fellows, at a nominal cost, typewritten extracts and transcriptions from journals which are either possessed by it in its library or are available elsewhere in Calcutta.

The *Society* awards research grants in pursuance of its objective to promote chemical research in the country. Due to financial stringency, however, the awards have been kept in abeyance at present. The *Society* awarded during the years 1933-38 the Sir P. C. Ray 70th Birthday Commemoration Medals to research workers selected among the junior chemists on the basis of their merit. In memory of the late Mr. J. M. Das Gupta, a Fellow of the *Society*, the award of a second gold medal has been instituted since 1934.

## International Civil Aviation Organization (I.C.A.O.) South-East Asia Regional Conference

THE South-East Asia Regional Air Conference held under the auspices of the International Civil Aviation Organization was inaugurated by the Prime Minister of India, Pandit Jawaharlal Nehru, on November 16, 1948 at New Delhi. Some 225 delegates and representatives from 16 countries attended the Conference. The countries represented were: Burma, India, Indonesia, Iran, the Netherlands, Pakistan, Siam, United Kingdom, United States of America, France, Belgium, Phillipines and Australia. Several members of the Secretariat, I.C.A.O. were also present. Dr.

Edward Warner, President of the Council of the I.C.A.O., was present to guide the work of the Conference. The presence of the delegates and experts from countries in other regions is evidence of the great significance attached to the Asian continent in the future of world aviation.

Pandit Nehru in his address spoke of the remarkable progress of aviation in India and its major rôle in the history of mankind. This rapid progress, he said, was natural because India is ideally placed and has vast areas where air travel becomes almost necessary and essential. The climate for the

great part of the year was suitable for aviation. He emphasized the central position India occupied in the geographical set-up of South-East Asia and the world. The I.C.A.O. which enjoys the actual support of 51 nations of the world, in the words of Dr. Edward Warner, "is an association of National Governments which have recognized the need for working together for the good of civil aviation and for the healthy development of international relationships". The present Conference, which is the ninth of the first series of regional meetings, is to examine the problems of air navigation in South-East Asian region and prepare a regional plan of aids to navigation and usages.

An idea of the development of civil aviation in India during the past 2 decades may be had from the fact that while the total expenditure on civil aviation during 1947-48 was Rs. 10,11,000, the figure for 1948-49 is expected to be Rs. 6,22,82,000. The rapid strides in civil aviation has been mainly due to the expeditious way the 10-year post-war plan drawn up some 3 years ago has been implemented. According to this plan, the Civil Aviation Department would maintain and develop the already existing 45 aerodromes in India; 21 aerodromes in the Indian States are being maintained and developed by the States concerned. In addition, the plan provides for the construction of 21 new aerodromes mainly for local communications. The aerodromes at Santa Cruz (Bombay), Dum Dum (Calcutta) and Palam (New Delhi) are being developed with a view to bring them to the standard of international aerodromes.

Training facilities for pilots, engineers and other technical personnel to man civil aircraft in India are available at 7 subsidized flying clubs at Bombay, Madras, Delhi, Barrackpore, Patna, Bhubaneswar and Lucknow, and at 3 other non-subsidized clubs at Jodhpur, Trivandrum and Hyderabad. 3 more are being planned at Jullundur, Nagpur and Gauhati during 1949. The Communication School at Saharanpur is training radio-operators and technicians and a new school is to be established at Allahabad for training students in flying, flying control and administration. The Government of India, in this connection, have **deputed 6 experienced**

pilots for training in the United Kingdom as instructors. The Allahabad school is expected to turn out 300 control operators during the next 3 years.

During the first two quarters of 1948 there was a 25 per cent increase in air-traffic as compared with the preceding half year, the miles flown being 58,74,380 and the total number of passengers carried were 1,75,734. A 27 per cent increase in the weight of freight mails and newspapers carried was recorded—the figures being: air freight, 1,421 tons; newspapers, 743 tons; and in ils, 286 tons. An improvement of 3.8 per cent in the factor of regularity was recorded which was 99.7 per cent—a record for India. The number of aircraft registered rose from 551 to 614 and the number of B class pilots increased to 296, A class pilots to 333, A-1 class to 15 and ground engineers to 343.

The Conference elected Rai Bahadur N. C. Ghosh, Director-General of Civil Aviation in India, as *Chairman*. Mr. Md. Ismail, leader of Pakistan delegation and M. Hague-nan, leader of the French delegation, were elected first and second *Vice-Chairmen*.

The Fact-finding Committee, which met earlier during the month, submitted its report to the Conference on the basis of which the Conference will formulate its decisions. The Committee assembled factual data relating to air routes, aerodromes and ground sites, air traffic control, communication, meteorology and search and rescue organizations for South-East Asian region comprising of Afghanistan, Burma, Ceylon, Indonesia, India, Pakistan, Siam and portions of U.S.S.R., Australia and the Phillipines.

The Conference will study, in particular, the allotment of high-frequency radio channels for aircraft use. There is a great dearth of such channels and the International Tele-communications Union has asked the I.C.A.O. to prepare a plan for meeting the needs of international civil aviation with the greatest possible economy, having regard to the needs of civil aviation in all regions.

The Conference appointed 5 sub-committees to deal with aerodromes and air-routes, air traffic control, communications, meteorology and search and rescue. The allocation of radio frequencies was left to the consideration of the sub-committee on communications.



# Characteristics of the Ionosphere at Calcutta (October 1948)

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**T**HE following are the ionospheric data collected at Calcutta for the month of October 1948. The observations were made at each hour of the day for 5 days a week.

Fig. 1 presents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmission during January 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when the E region was found to be abnormal and the corresponding penetration frequencies and heights. The occurrence of abnormal E ionization at night is a feature of the ionosphere for this month.

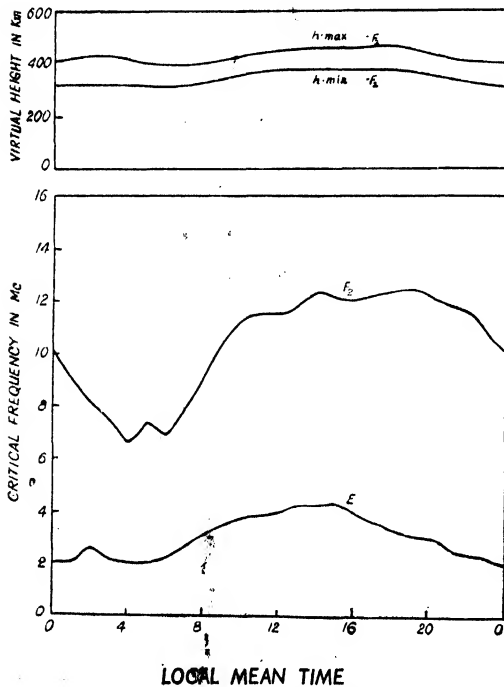
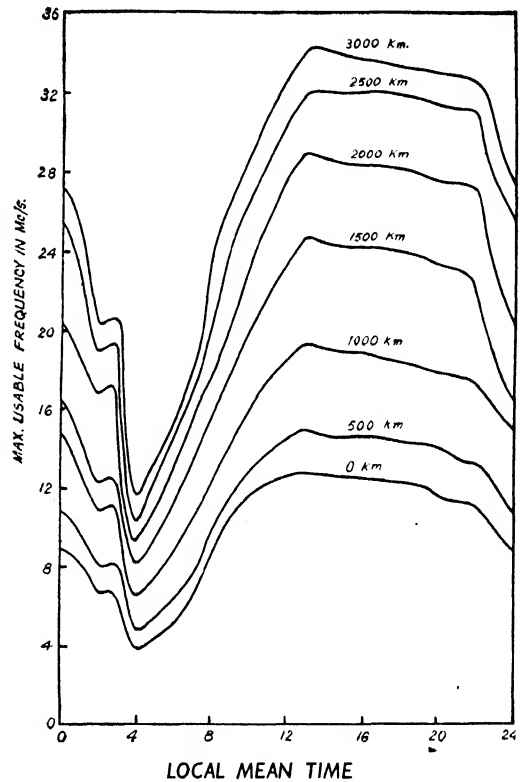


FIG. 1. — OCTOBER 1948.



AT POINT OF REFLECTION.  
FIG. 2. — PREDICTED M.U.F. FOR TRANSMISSION  
VIA  $F_2$  LAYER, JANUARY 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f_oF_2$ Mc.	$h'_{E_s}$ Km.
Oct. 1948	4	1800	4.00	120
		1900	3.75	120
		2000	4.25	120
		2100	5.70	120
		2200	6.10	120
		2300	5.70	120
	5	0000	5.25	120
		0100	4.00	120
		0200	4.55	120
		1700	4.20	120
		1800	3.85	120
		1900	3.70	120
		2000	3.55	120
		2100	3.25	120
		2200	3.00	105
		2300	4.00	105

TABLE I—*contd.*

MONTH & YEAR	DATE	Hour	$f^{\circ}E_s$ Mc.	$hE_s$ Km.
Oct. 1948	6	0000	5.00	120
		0100	4.80	120
		0200	3.00	105
	7	2200	5.45	120
		2300	5.05	120
	8	0000	4.95	105
		0100	4.50	105
		0200	4.25	105
		0300	4.00	90
		0400	3.05	90
	9	2300	3.20	105

TABLE I—*contd.*

MONTH & YEAR	DATE	Hour	$f^{\circ}E_s$ Mc.	$hE_s$ Km.
Oct. 1948	10	0000	4.50	105
		0200	4.65	105
	21	1400	4.75	135
		1500	5.90	135
		1600	6.20	135
		1700	6.30	135
		1800	6.45	135
		1900	6.55	135
		2000	7.00	135
		2100	7.00	133
		2200	6.85	135
		2300	6.65	120
	22	0000	5.90	120
		0100	4.10	120
		0200	3.90	105

## Utilization of Chlorine

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IT is estimated that about 54,000 tons of caustic soda are needed in India annually for various industrial purposes<sup>1</sup>. If produced electrolytically, this would result in the simultaneous production of about 48,000 tons of chlorine. Large quantities of chlorine will thus become available in India for industrial purposes as soon as new plants for production of caustic soda (some of which are now under erection) start functioning. Fears have been expressed that such quantities of chlorine may not find markets in India.

Chlorine is an important industrial chemical, and an increase in the production of chlorine should be welcomed by everybody interested in the development of chemical industries. The importance of chlorine as a raw material will be obvious from the fact that in 1940, in America<sup>2</sup> alone, production of chlorine rose to 5,00,000 tons a year. This quantity found consumption as follows:

- |  |    |
|--|----|
| 1. For the manufacture of chlorinated hydrocarbons, solvents, glycols, chlornaphthalenes, etc. | 60 |
| 2. For bleaching operations in   |    |
| (a) Paper industry   | 21 |
| (b) Textile industry   | 5  |
| 3. Sanitation  | 6  |
| 4. Other uses  | 8  |

During the war large quantities of chlorine were consumed by the military for the production of smoke screens and for various chlorine compounds used in chemical warfare.

The above analysis may be found instructive as it indicates the spheres in chemical industries which consume large quantities of chlorine in other countries. It is noteworthy that 60 per cent of the total quantity produced in America was consumed in the manufacture of chlorinated hydrocarbons and allied solvents, a fact that should be helpful in planning for the consumption of chlorine that is expected to be available in our country.

Almost the entire quantity of chlorine that is produced at present is consumed in the manufacture of bleaching powder or of bleach liquors. Some quantities are consumed in the treatment of water and for sanitation purposes, but little attention has so far been paid by Indian manufacturers to the numerous chlorine compounds that can be manufactured with chlorine and other materials which either exist or can be produced in India. It is proposed to consider the possibilities for the utilization of chlorine with this end in view in this paper. For the sake of convenience,

production of chlorine compounds may be considered under the following heads :

- (1) Chlorohydrocarbon solvents.
- (2) Chlorine compounds needed for military operations.
- (3) Chlorine compounds ( inorganic ) needed for industrial purposes.
- (4) Chlorinated oils, resins and rubber.

#### Chlorohydrocarbon Solvents

Production of cheap solvents in India may result in rapid developments in various fields of chemical industry. For instance, modern methods for the extraction of vegetable oils from oil seeds are based on solvent action, and developments in oil mills may soon follow the availability of cheap solvents. Use of solvents has led to remarkable developments in lacquer industry in other countries, and old methods involving the use of linseed and other vegetable oils in lacquer and paint industries in India may have to give place to different methods as soon as solvents are available at economic prices. Extraction of essential oils, of alkaloids and active principles of drugs, preparation of insecticides, degreasing of bones and metals and dry-cleaning operations are other instances of industrial processes which may develop rapidly if solvents are available.

According to McGovern<sup>3</sup> the following chlorohydrocarbons are manufactured in America, and find use as solvents in industries : methyl chloride, methylene chloride, chloroform, carbon tetrachloride, dichloroethylene, trichloroethylene, perchlorethylene, ethyl chloride, ethylene dichloride, 1, 1, 2-B trichlorethane, 1, 1, 2, 2, tetrachlorethane, pentachlorethane, hexachlorethane and propylene dichloride. In India, however, the production of solvents has so far been very limited on account of the difficulties in the availability of raw materials. The following chlorohydrocarbons may be manufactured in India from chlorine and hydrocarbons that are either available or can be produced from alcohol. The materials that will be needed for the production of chlorohydrocarbons are mentioned against each :

Solvents	Raw materials
Carbon tetrachloride	Chlorine and natural gas
Chloroform	Chlorine and alcohol
Ethylene chlorohydrin and glycols	Chlorine and ethylene
Trichlorethylene and perchlorethylene	Chlorine and acetylene ( or alcohol )
Dichlorethane	Chlorine and ethane

Natural gas occurs in India, and ethylene, ethane and acetylene can be produced from alcohol. Processes for their manufacture from alcohol are simple, but no distillery in India has so far attempted to manufacture these hydrocarbons. The raw materials needed for the production of the above solvents, references to methods of their production, and their important industrial applications are briefly as follows :

(a) *Carbon Tetrachloride (Raw material : Natural Gas)* — Natural gas has been tapped at Jagatia and Gogha in Kathiawar and at Baroda<sup>4</sup>. In smaller quantities it is reported from wells in Saharwel<sup>5</sup> ( Mymensingh District ). According to Barton<sup>6</sup> who investigated the resources for natural gas in Burma, on Indaw oil-fields alone 12 million cu. ft. of gas are produced per day, and in Thryetmyo District, gas is liberated at a depth of 2,525 ft. in quantities estimated at 39 million cu. ft. per 24 hr.

If natural gas is collected from these fields and chlorine from the new caustic soda chlorine plants, production of carbon tetrachloride may be started according to the methods described below. Sources of natural gas in India are considered poor, but it may be pointed out that the requirements are such that even if 10,000 cu. ft. of gas are available per day, an economic unit can be set up.

Tolloczko and Kling<sup>7</sup> prepared carbon tetrachloride by chlorinating natural gas at 400°C. in the presence of catalysts. Jones, Allison and Meigham<sup>8</sup> chlorinated natural gas containing 88.5 per cent methane, 10.1 per cent ethane, 0.4 per cent nitrogen. Baskeville Reiderer<sup>9</sup> chlorinated natural gas containing 50-90 per cent methane by subjecting the mixture to silent electric discharge. Tizard, Chapman and Taylor<sup>10</sup> passed methane over pumice impregnated with copper chloride at 435°C. and obtained chlorinated hydrocarbons with carbon tetrachloride and chloroform as the main products.

**Technical Applications** — Carbon tetrachloride is known industrially under different names — "Tetra", "Tetracal", "Asordin", "Phoenixine" are some of the trade names under which it is sold in large quantities in America and in the U.K. One of the most important applications of carbon tetrachloride is as a fire extinguisher, particularly for fires involving volatile organic solvents. Industries which consume large quantities of tetrachloride include petro-

leum refineries ( where it is used as a solvent for gasoline, paraffin and petroleum ), and rubber factories where it is consumed as a constituent of solvents. During the war large quantities of carbon tetrachloride were needed for the manufacture of smoke screens. Other industrial operations for which carbon tetrachloride may be considered as a solvent are (a) dry cleaning ; (b) degreasing of bones in glue plants ; (c) extraction of atropine, strychnine and other alkaloids in pharmaceutical works ; and (d) degreasing of metal parts preparatory to plating, galvanizing, etc. It may also be used as a refrigeration medium, as an ingredient in shoe polishes, and as a constituent in the preparation of insecticides. The demand for a cheap solvent, particularly for dry cleaning and degreasing, has increased considerably during recent years.

(b) *Chloroform (Raw material: Alcohol)* — Alcohol is produced in large quantities in India, and its production is likely to increase in the future. The industrial importance of some of the compounds that can be produced from alcohol (e.g. acetone, ether, hydrocarbons, acetic acid, etc.) is so great, that it may be more profitable for some of the distilleries to utilize the alcohol for the production of these industrial chemicals.

When alcohol is distilled with bleaching powder, chloroform is produced. The method is an old one and is still used. Acetone is now more commonly used in other countries, but in view of the fact that Indian distilleries produce alcohol, the older method may be advantageous. A variation consists in chlorinating absolute alcohol with anhydrous chlorine gas and treating the chloral hydrate thus formed with alkali<sup>11</sup>.

Production of chloroform from alcohol should be a matter of special interest for such firms as have their own distilleries and are planning for installation of caustic soda plants.

**Technical Applications** — Like carbon tetrachloride, chloroform is extensively used for the extraction of oils, fats and waxes ; and for degreasing bones, metal surfaces, furs and wools. One of its most important uses is in the pharmaceutical industry where, besides its well-known use as an anaesthetic, it is employed as a constituent of antiseptic preparations, inhalants, lineaments, and as a preservative for various vegetable drugs. With carbon tetrachloride and gasoline, it finds use in dry-cleaning operations. Paper

mills consume large quantities of chloroform for degreasing paper stocks. Chloroform has recently been used in the manufacture of several food preparations, particularly in making food flavours and in the manufacture of vitamins. During the war, large quantities of chloroform were consumed in the manufacture of hexachlorethane needed in the production of smoke screens.

(c) *Ethylene Chlorohydrin & Glycols (Raw material: Ethylene)* — Production of ethylene from alcohol is a simple process. Number of catalysts have been developed that effect almost a quantitative conversion of alcohol into ethylene. Sprent<sup>12</sup> described the production of ethylene by passing alcohol vapour over amorphous alumina at 310°C. According to Sanderens<sup>13</sup>, the following catalysts have proved highly efficient :

CATALYST	TEMPERATURE °C.	% ETHYLENE IN GAS
Al. phosphate	320	99.5
Al. silicate	270	99.5
Kaolin	270	97.5
Al. sulphate (anhydrous)	265	99.5
Ppt. alumina	250	99.5

Ethylene, ethane and acetylene are raw materials for many important industries and in the absence of large quantities of natural gas, their production from alcohol deserves every consideration from those interested in the planning of new industries.

The production of ethylene chlorohydrin and glycols can be accomplished according to the following processes :

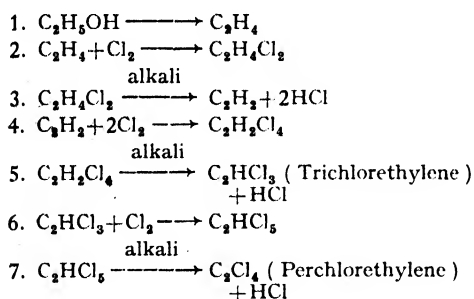
Chlorine and ethylene are agitated in water. The solution is neutralized with lime and ethyl chlorohydrin distilled over<sup>14</sup>. According to a British Patent<sup>15</sup>, water is circulated through a tower where it is treated with chlorine gas, forming a solution of hydrogen oxychloride and hydrogen chloride. It then passes to a second tower where it comes into contact with ethylene which at 0°-20°C. combines with hydrogen chloride to form ethylene chlorohydrin. The latter decomposes on boiling with water directly into glycols which find numerous applications as industrial solvents.

(d) *Trichlorethylene & Perchlorethylene (Raw materials: Alcohol [Ethylene & Acetylene])* — Starting with 90 per cent industrial alcohol, it is possible to manufacture trichlorethylene and perchlorethylene, two chlorohydrocarbons which have acquired considerable importance as solvents. According to the scheme for their manufacture

described below, chlorination is involved at three stages.

Production of ethylene from alcohol for industrial purposes is well known, but that of acetylene (which is really the raw material for the above solvents) is usually carried out from calcium carbide. Carbide is not manufactured in India at present, but the Heavy Chemicals and Electrochemical Industries Panel have recommended<sup>16</sup> 7,000 tons as the target, and the Government of India have granted an import licence for a 5,000-ton plant to be installed in Bihar. The absence of this basic industry has retarded the development of a large number of chemical industries in India. When the carbide industry is established, the manufacture of trichlorethylene and perchlorethylene will be easy, as the first three stages, which represent production of acetylene from alcohol may in that case be completely eliminated. But as long as carbide is not available, production of these solvents may be considered from alcohol.

The series of chemical reactions that are involved in the production of trichlorethylene and perchlorethylene from alcohol (or acetylene) and chlorine are briefly as follows :



**Technical Applications**—Trichlorethylene is mainly used for the extraction of oils and fats, and for degreasing fish scrap, textiles, and metals. In India, trichlorethylene is likely to find use in dry cleaning, oil mills, paper mills, electroplating, and pharmaceutical works.

Perchlorethylene is sold in other countries under the names "Etdine", "Perwin", and "Tetralex". Its main uses are : (i) in textile mills as an ingredient of compositions used for waxed cloth, for treating fibres and fabrics for producing various designs ; and (ii) in the manufacture of photographic films where it is used as a solvent for cellulose acetate, or nitro-cellulose. During the war

large quantities of perchlorethylene were used for the manufacture<sup>17</sup> of smoke screens.

(e) *Dichlorethane (Raw material: Ethane)*—Like ethylene, ethane is produced by the catalytic dehydration of alcohol. Boomer and Morris<sup>18</sup> obtained ethane by the catalytic decomposition of alcohol at 300°-500°C. with silica gel. When heated at 615°C. at a pressure of 170 atm. in the presence of aluminium-copper catalysts, the proportion of ethane in the resulting gases was found to be as high as 74.8 per cent<sup>19</sup>. When ethane and chlorine are led over activated charcoal at 100°-300°C., dichlorethane is produced<sup>20</sup>.

Dichlorethane is extensively used as a solvent for rubber. It readily dissolves bakelite, camphor, rosin, vinyl and glyceryl phthalates resins, bees-wax and carnauba wax. It has found numerous applications in the manufacture of plastics and varnishes, as a medium in refrigeration plants, and as a solvent in dry-cleaning operations.

#### Chlorine Compounds in Military Operations

Two types of chlorine compounds have been used during the war : (i) those used in the preparation of smoke screens ; and (ii) those used for the manufacture of lachrymatory agents, vesicants and toxic gases.

(i) *Smoke Screens*—Berger mixture, popularly known as "BM", consists of carbon tetrachloride 41 per cent, zinc dust 35.4 per cent, sodium chlorate 9.3 per cent, ammonium chloride 5.3 per cent, and magnesium carbonate 8.3 per cent<sup>21</sup>. This mixture was used in large quantities in World War I. Improvements over BM have since been reported. The H.C. mixture consists of solid hexachlorethane 50 per cent, zinc dust 28 per cent, and zinc oxide 22 per cent. Hexachlorethane, the chief constituent in the mixture, is prepared either by heating chloroform in a red-hot tube<sup>22</sup>, or by heating carbon tetrachloride to 160°C. with arsenic<sup>23</sup>.

Another type of smoke screen is represented by titanium tetrachloride and silicon tetrachloride. Tin tetrachloride has been used in certain countries, but as tin is not available in India in any considerable quantity, it may not be possible to manufacture this tetrachloride on a commercial scale. Titanium tetrachloride ( $\text{TiCl}_4$ ), popularly known as "FM", can be manufactured in India when required. The process consists in mixing titanium ore with carbon and heating the mixture to about 650°C. in a current of chlorine.

Silicon tetrachloride ( $\text{SiCl}_4$ ) is prepared by treating silicon carbide with chlorine.

(ii) *Lachrymatory & Vesicant Agents & Toxic Gases:*

(a) Chloroacetone,  $\text{CH}_3\text{CO}\cdot\text{CH}_2\text{Cl}$ , also known as "Tonite" is produced by passing chlorine through acetone. It lachrymates the eyes in concentrations as low as 0.018 mg. per litre<sup>24</sup>.

(b) Chloropicrin,  $\text{C}\cdot\text{Cl}_3\cdot\text{NO}_2$ , also known as "Aquinite", "Klop", "PS" and "NC", produced by the action of chlorine on picric acid or its salts, induces lachrymation at as low a concentration as 0.002 mg. per litre; and at 2.00 mg. per litre it is lethal.

(c) Chloroacetophenone,  $\text{C}_6\text{H}_5\cdot\text{CO}\cdot\text{CH}_2\text{Cl}$ , known under the name "CN", is prepared by chlorinating acetic acid, followed by chlorination of chloroacetic acid in the presence of sulphur chlorides. The chloroacetyl chloride is finally treated with benzene in the presence of anhydrous aluminium chloride.

Chloroacetophenone is solid and resistant to heat and moisture, and on account of these qualities it was used in large quantities during the last war. Its production in India will depend on the availability of benzene and acetic acid. Benzene is produced by certain coke works in Bihar, but acetic acid is not manufactured in sufficient quantities in India at present.

(d) Mustard Gas — According to the process that was employed by the Germans in Great War I, mustard gas is obtained from ethylene chlorhydrin.

The production of ethylene from alcohol on an industrial scale has already been considered. For distilleries producing ethylene, the production of mustard gas should be possible.

(e) Chlorvinyl dichlorarsine — This compound was developed in America at the close of World War I and is described as "America's principal contribution to the *Materia Chemica* of the World War". This was an improvement over mustard gas or ethyldichlorarsine, and large quantities were manufactured during the war. The main raw materials were acetylene and arsenic chloride.

The production of acetylene from chlorine and alcohol has been discussed in connection with the preparation of chlorohydrocarbon solvents. Arsenic, the other raw material, occurs in India. Deposits of orpiment and realgar are known in Shankalpa glacier in

Kumaon<sup>25</sup>. Mallet<sup>26</sup> has described the occurrence of arsenical pyrites on Samphar hills (Darjeeling). Arsenic is also obtained as a by-product in the metallurgical treatment of several ores. Thus, if necessary, it would be possible to take up the production of such dangerous chemicals as ethyldichlorarsine and chlorvinyl dichlorarsine and other compounds of the series.

**Chlorine Compounds (Inorganic)  
Needed for Industrial Purposes**

The demand for bleaching powder from the paper and textile mills in India has increased considerably during recent years. Since lime, which is the only other raw material needed for the production, is available, the manufacture of bleaching powder has been started in most of the factories producing chlorine. Potassium chlorate can be manufactured from lime and potassium carbonate. Sulphur chlorides, mainly used in the rubber industry, may be manufactured with imported sulphur even as sulphuric acid or other sulphur compounds are being manufactured at present. Chlorides of tin, mercury and phosphorus are produced by the interaction of chlorine with the respective elements, but these are not produced in India. It may, however, be noted that chlorides of tin, phosphorus and mercury are important materials, and their production, even from imported materials, may be considered on account of the large number of new industries that are likely to develop if these chemicals are produced.

Chlorination of minerals has led to the production of important salts of metals on a commercial scale. Maier<sup>27</sup> chlorinated chromite ore (mixed with carbon and quartz, and reduced to 100 mesh) at a temperature of 900°C. On sublimation chromium chlorides were obtained. India produces about 60,000 tons of chromite annually<sup>28</sup>, and only a part of this is worked up for the production of chromium compounds. The chlorination process of Maier may be adopted for production of chromium salts required in the tanning and plating industries and for the manufacture of chromium pigments.

The chlorination of pyrites has been investigated by Ezzakov<sup>29</sup> who used pyrites containing 49 per cent sulphur and obtained 82 per cent conversion. Ezzakov has shown that the chlorination resulted in the production of ferric chloride and sulphur chloride both of which are compounds of

considerable industrial importance. It may be noted in this connection that quantities of iron pyrites exist in India particularly in Simla hills, and they have not been utilized so far, as their use in the manufacture of sulphuric acid has not been found economically feasible. According to Pristonfil<sup>30</sup>, treatment of zinc blende with ferric chloride results in the formation of zinc chloride and separation of sulphur. Zinc ores are known to occur near Udaipur in Rajputana. Combining, therefore, the results of Ezdakov and Pristonfil, it may be possible to utilize chlorine (from alkali plants), iron pyrites (from Simla hills) and zinc blende from Udaipur, and work out schemes for the production of sulphur chlorides, zinc chloride, iron chloride and sulphur. The industrial importance of these chemicals is well known.

Chlorination of titanium ores has been investigated by a number of workers. The methods employed consist in mixing the ore<sup>31</sup> with 30 per cent carbon, and heating the mixture to about 650°C. A fused mass consisting of titanium carbonitride ( $Ti_5C_4N_4$ ) and titanium carbide is formed which can be converted into  $TiCl_4$  by heating in a current of chlorine. Muskat and Tylors<sup>32</sup> have described an apparatus for the preparation of titanium chloride by a continuous process, in which conditions are so regulated that chlorine and the ore react at 600°C., attained as a result of the heat of reaction. In another process described by Taggent<sup>33</sup>, briquettes are made by bonding rutile and charcoal, and chlorinated at 700°C. The condensate obtained contains titanium tetrachloride 99 per cent, silica 0.5 per cent, and iron 0.002 per cent.

Chlorination of bauxite has been investigated by Finke and Marchi<sup>34</sup>, who worked out details of a process for the production of aluminium chloride from bauxite.

#### Chlorination of Oils, Resins & Rubber

Considerable work has been done on the chlorination of mineral oils in other countries, but oils of mineral origin may not be available for the preparation of chlorine compounds in India. Vegetable oils are available, but their chlorination has not led to any useful results so far. Chlorination of animal fats in some cases has yielded valuable products. Whale oil, on chlorination, yields products which according to a British Patent<sup>35</sup> are used as bonding agents for abrasives, plastics, rubber goods and

composition leather substitutes. Chlorinated sardine oil has found use in germicidal preparations. Fish oil is produced in large quantities in Madras and Bombay, and its industrial possibilities have not been fully explored. It may be mentioned in this connection that owing to the scarcity of mineral oils in India, difficulties are experienced in finding substitute oils which may be used for larvicidal purposes, and chlorinated fish oils may be investigated as possible substitutes for mineral oils for anti-malarial operations.

Chlorination of hydrocarbons has resulted in the development of a large number of resins of industrial importance. Chlorinated diphenyl resins or "Arochlors", for instance, are obtained on chlorination of diphenyl. They are extensively used for the manufacture of insulating compositions. Tung oil or linseed oil varnishes prepared with "arochlor" are known to be highly resistant to water and alkalies. Wood and textiles when treated with "arochlors" are rendered flame-proof.

Diphenyl, the only material needed for this preparation, is not manufactured in India, but its production from benzene is simple enough. According to certain American Patents<sup>36</sup> benzene vapour when led at 600°-800°C. through a lead bath is converted into diphenyl. Magnetic oxide of iron is used as catalyst at 800°-950°C. in large-scale manufacture.

Chlorination of naphthalene results in the production of a mixture of chloronaphthalenes with a melting point ranging from 60°-125°C. "Seekay" wax (I.C.I.), and "Halowax" are two well-known chlorinated naphthalenes. On account of their non-inflammability, chloronaphthalenes are used as binding agents in the manufacture of condensers, and for insulating and sealing compositions for electric goods. During the war, chloronaphthalenes were in great demand for the production of smoke screens and for the damp-proofing of explosives. Emulsified preparations of some of the chloronaphthalenes are extensively used as insecticides<sup>37</sup>. In recent years chloronaphthalenes have found use as substitutes for camphor in the production of nitro-cellulose films, and in the production of polishes. The manufacture of chloronaphthalenes is, therefore, likely to be a profitable industry in India. There are other chlorinated hydrocarbons which have found industrial applications in other countries (e.g. resins from chlorinated cymene);

but they are not likely to be of importance in India on account of the difficulties involved in obtaining suitable hydrocarbons.

**Chlorinated Rubber** — Raw rubber is produced in Madras, Mysore, Travancore and Cochin. During 1936-40 India exported 23,327,653 lb. of raw rubber<sup>38</sup>. When chlorine is passed into solutions of raw rubber, chlorinated resins employed in the preparation of wrapping sheets, moulded products, etc., are obtained. Varnishes produced from chlorinated rubber have been extensively used as coatings for aircraft and automobiles. "Tonsite", a chlorinated product used in Germany during the war, is a non-inflammable powder highly resistant to the action of acids, alkalis or salts. Transparent sheets, laminated products, and waterproof cloth have been obtained by Karimullah and Uma Shankar<sup>39</sup> by chlorinating cyclized rubber.

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## Indian Standards for Refractories

THE *Engineering Division Council* OF THE *I.S.I.* has brought out three Draft Indian Standards on Fireclay Refractories. They are concerned with Moderate Heat Duty Fireclay Refractories, Group 'A'; Moderate Heat Duty Fireclay Refractories, Group 'B'; and High Heat Duty Fireclay Refractories. These have been prepared by an expert Sectional Committee composed of representatives of manufacturers and consumers of refractories in India. The Committee is headed by Dr. H. K. Mitra of the *Tata Iron & Steel Co. Ltd.*, Jamshedpur.

The Drafts consist of standard specifications for different types of refractories

produced in India, and the methods for chemical analysis, determination of pyrometric cone equivalents under load for full-size bricks as well as for sections of bricks, the porosity, and resistance to spalling. The object of the specifications and tests is to provide an agreed method of evaluation for the three different types of refractories.

These Drafts on fireclay refractories have been widely circulated to industrialists and technologists in the field. Comments will be received till 29th February 1949 by the Director, *Indian Standards Institution*, 'P' Block, Raisina Road, New Delhi.



# Scope for the Cultivation of Medicinal Plants in India

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(Continued from Dec. 1948 issue)

## 11. *Claviceps purpurea* Tulasne (Ergot)

THE sclerotium of this fungus arising in the ovary of rye, *Secale cereale* Linn., is known as "Ergot".

The drug is specially cultured in Russia, Poland, Spain and Portugal. It is the only oxytocic or uterine tonic drug known which is effective on administration by the oral route. It is largely used in India where proper medical aid at child-birth is often beyond the reach and means of the bulk of rural population.

It is observed by R. Tyller<sup>8</sup> that barley in the upper province of India was often affected with a disease similar to, if not identical with, ergot. The same or apparently the same disease was observed on rice and oats during recent years. No effort was, however, made to develop ergot from Indian sources and the entire supply comes from Europe.

A good deal has been written on the poisonous properties of barley, wheat and millet which, to outward appearance, seem to be of good quality but which contain a fungus most probably resembling ergot. Recently a fungus, most probably ergot, was observed on the grasses in Simla hills<sup>9</sup> and on sugarcane and on *Cynon dactylon* in Mysore State. This has been confirmed.\* In fact ergot has been successfully produced in India.

Ergot production was taken up by Mr. M. K. Thomas<sup>10</sup> in 1942 in Madras. The exploratory field experiments were conducted over 2 acres of rye raised in the Government Research Station in the Nilgiris. The

botanical, chemical and pharmacological examination of ergot grown in the Nilgiris satisfies all the requirements laid down in the *British Pharmacopoeia*<sup>11</sup>.

The water-soluble alkaloid of ergot, ergometrine and the water-insoluble ergotoxine-ergotamine present in Indian ergot have been determined. The results are as follows:

Total alkaloids	% 0.1213
(calculated as ergotoxine)	
Water-insoluble alkaloids	0.1169
(calculated as ergotoxine)	
Water-soluble alkaloids	0.237
(calculated as ergometrine)	

**Pharmacological Examination** — Using Broom and Clark's method of assay with rabbit uterus, the ergotoxine content in the liquid extract (*B.P.* method) lies between 0.085 and 0.145 per cent (mean, 0.115 per cent). This would not probably be far from the true value of ergotoxine content in the liquid extract.

It is evident that ergot artificially produced on rye in India is of good quality. It is in certain respects better than many of the imported batches of ergot.

Since the exploratory field experiments on the production of ergot in the Nilgiris have proved successful and ergot of standard quality has been produced, the Director of Agriculture, Madras, prepared a scheme for the production of ergot on a large scale. The following extract from the personal communication of the Government Mycologist of Madras Presidency show the pro-

**Transverse Section** — The outer portion consists of small dark-coloured cells, the colour of which is changed to brownish red on the addition of sulphuric acid. The rest of sclerotia consists of nearly colourless, closely compacted, very small oval or round cells. Imported sclerotia of ergot also shows more or less similar appearance, odour and taste which are characteristic.

**Chemical Examination** — Total alkaloids of ergot 0.13014 per cent (130.14 mg. per 100 gm.) *British Pharmacopoeia* specifies 0.05 per cent (50 mg. per 100 gm.).

\* **Botanical Examination** — Length of sclerotia — 2 to 3 cm.; smallest size 1 cm. Some sclerotia are cylindrical with a thick base and nearly pointed tip, others are markedly curved. Appearance, dark-coloured hard structures 4 to 5 mm. thick with a yellowish core. The length of the sclerotia imported from Europe varies from 1 to 3 cm. These are nearly cylindrical, slightly curved with longitudinal furrows, extensively dark brown with a pinkish core.

gress of the culture and production of ergot carried out in the Nilgiris :

"Ergot of rye is being produced on the Nilgiris from 1943 onwards. Rye is specially grown for this purpose, as it is not one of the cereals cultivated by the local people. The total area put under rye for this purpose does not exceed 100 acres.

"The production of ergot is at present limited to meet the requirements of the province and the surplus, if any, is offered for sale to private pharmaceutical firms. The target of production is at present kept at 2,500 lb. of ergot per annum. But the locality is well suited for large-scale production of ergot to meet the entire requirements of India as it has favourable climatic conditions.

"The quality of the ergot is being improved progressively by continuous selection of the strains exhibiting high alkaloid contents. Thus it has been possible to raise the total alkaloid content of this bulk produce from 0.19 per cent to 0.4 per cent calculated as ergotoxine. Further work on these lines is in the progress."

Shillong (Assam) is also suggested to be a suitable place for culturing ergot<sup>12</sup>. It enjoys a temperate climate and heavy rainfall. The physical features and some of the meteorological conditions of Shillong compare well with those of New South Wales in Australia where, during recent years, culture of ergot has attained remarkable success. Besides Shillong experiments for culturing ergot may be undertaken in Simla Hills and Mysore State where locally growing grasses have shown to be infected with a similar fungus.

12. *Digitalis purpurea* Linn. (Foxglove) — This plant is a native of Western Europe but is now being extensively cultivated in many parts of the world, particularly in England, Germany, Austria, etc. *Digitalis* leaves are largely used as a heart tonic. Large quantities of *digitalis* preparations used in India are imported from abroad. It has been observed that preparations of *digitalis*, especially the tincture, lose 20 to 40 per cent of their potency within a few months under climatic conditions prevailing in India and are rendered useless for therapeutic use.

So far as is known, none of the species of *digitalis* is indigenous to India. The cultivation of *Digitalis purpurea* was started on an experimental scale many years ago. Regular crops now grow in some of the hill

stations, particularly in Kashmir. The Forest Department of Kashmir State is cultivating the plant on a large scale at Tangmarg and in Kishtwar. Regular crops of leaves are obtained every year which, on biological assay, have been found to be of excellent quality.

*Digitalis* was also grown in Mungpoo near Darjeeling and in the Nilgiris, but as the leaf matures in these places during the rainy season, drying and curing of the leaf is difficult under natural conditions; artificial means of drying, e.g. by heat involves the risk of the glucosides being decomposed and becoming toxic. Tinctures prepared from the leaves obtained from different places were biologically assayed and clinically tried<sup>13</sup> with good results.

The tinctures made from Darjeeling leaf varied a great deal in their activity as revealed by biological assay and clinical trials; they were not nearly so good as tinctures made from the Kashmir leaf. The tinctures made from the Nilgiri leaf were toxic, and were not therapeutically effective. The leaf grown in the Nilgiris could possibly be improved by better methods of cultivation and drying.

*Digitalis* grows well in open spaces at altitudes of 6,000' above sea level. Suitable localities can be selected in the Western Himalayas and the plant can be cultivated with success. Its cultivation on a large scale in India has a good future.

*Digitalis lanata* Ehrh. also grows well in Kashmir and leaf of excellent quality is obtained. This species is preferred by many as it is less cumulative in action, rapidly slows the pulse and its glucosides are more stable. Its preparations, therefore, deteriorate less in the tropical climes than *D. purpurea*. This variety grows well in Tangmarg (Kashmir) at an altitude of 7,000' above sea level and it could be cultivated in other suitable localities in India.

13. *Ephedra Gerardiana* Wall. — The official source of ephedrine is the plant *Ephedra sinica* Stapf. and *Ephedra equisetina* Bunge. and other species which are indigenous to China. *E. Gerardiana* Wall. and *E. nebrodensis* Tineo also contain the alkaloid and these plants are indigenous to India both in North-west Himalayas and in the Sikkim area. Many species of *ephedra* grow in Northern India, e.g. in Bushahr Division, Chakrata, Kangra, Kulu, Baluchistan, Kashmir, Hazara, Kagan, Trans-Frontier territory and Waziristan. Specimens from various places

have been analysed and it has been shown that ephedras growing in the drier regions of North-west India contain a high percentage of alkaloids; in many cases higher than the alkaloidal contents of the Chinese species recorded by Read and Feng<sup>1</sup>. Among the Indian species *E. nebrodensis* is the richest and *E. intermedia* the poorest so far as its ephedrine content is concerned. The Indian species also contain a larger percentage of pseudo-ephedrine than those growing in China.

In the present political set up of the country, India has limited sources of ephedra falling short of estimated requirements. Ephedra grows in the proximity of Kashmir valley only in very small quantities. It is, therefore, necessary either to extend its cultivation in the areas where it is known to grow, or to introduce it into suitable localities in Northern Himalayas where the plant is reported to be growing in a state of nature.

14. *Eugenia aromatica* (Linn.) Baill. (Clove) — The dried flower buds of this plant are known as "Clove". It is an ever-green tree indigenous to Malacca Islands and is cultivated in Zanzibar, Pemba, the Amboyan Islands, Penang, Madagascar and, to a lesser degree, in the Seychelles, Reunion, Mauritius and Ceylon. The plant has been cultivated in Government gardens in South India and Central Travancore with success<sup>8</sup>.

Cloves are aromatic, stimulant and carminative and are largely used in medicine and as a spice and flavouring agent also. They are used in perfumery and in the manufacture of "Vanillin". The demand for clove oil has increased, particularly, for aromatizing cigarette tobacco. 90 per cent of the world supply of cloves is obtained from two islands, Zanzibar and Pemba, where it forms the chief industry. India is one of the most important consumer of cloves and large quantities of clove oil are imported into India annually. The tree thrives in tropical climate and prefers volcanic soil in a sloping position and a certain amount of sand. It thrives in places with an annual rainfall of about 75" or more<sup>14</sup>. There is no reason why clove plantations should not flourish in South India if systematic cultivation is undertaken on scientific lines.

15. *Eucalyptus* — There are about 25 species of eucalyptus which yield the oil of commerce, chief amongst which are *E. globulus* Labill. and *E. dumosa* A. Cunn. Australia may be said to be the home of eucalyptus inasmuch

as it comprises 75 per cent of the vegetation of that continent. Eucalyptus oil is distilled from the fresh leaves and terminal branches of the tree and is an important article of commerce in this country. Large quantities of the oil are employed in scenting soaps and also in separating mineral sulphides from their ores by flotation. The oil is used in medicine for its powerful antiseptic and disinfectant properties. Of the constituents of the oil cineole (eucalyptol) is the most important ingredient from the medicinal point of view.

Eucalyptus trees are not indigenous to India but many species are grown in the Nilgiris. The tree is valuable on account of the essential oil and the dye, perfumes and kino which originate from it and these are all very valuable products. Attempts have been made during the last 50 years to cultivate it in many parts of the globe, e.g. California, Spain, South Africa, Algeria, East Africa, Mauritania, Java, Malaya. In India Eucalyptus was introduced before 1870 and the most successful results were obtained on the Nilgiri Hills where at present there are several large plantations covering an area of over 2,000 acres at altitudes of 4,000' to 8,000'. About 24,000 lb. of oil is distilled annually from these plantations which is not even a fraction of what the country needs. Large quantities are, therefore, imported.

The oil obtained from the leaves growing in the Nilgiri plantations was studied by Puran Singh<sup>15</sup>. It contains pinene, cineole, sesquiterpene and free alcohols in small amount. Phellandrene is present in the Australian oil in fair quantity; it is very irritating to the bronchial mucosa, especially when inhaled. Butyric and valeric aldehydes also are present in the Australian oil. Both these constituents are absent from the Indian oil and, therefore, it should be preferred for medicinal purposes. The constants of the oil are: specific gravity, 0.9065 to 0.9155; optical rotation, +5° to 10°; refractive index, 1.463 to 1.466; saponification value, 8.9 to 20; cineole 60 per cent.

The properties of the Indian oil compare favourably with the standards laid down in the *British Pharmacopoeia*, and the medical department of the Madras Government has used the Nilgiri oil with satisfactory results.

In Dehra Dun *E. terebaormis* and *E. creobra* have been cultivated, but the oil

obtained from their leaves does not come up to the standard of the *British Pharmacopoeia* and is not used in medicine. It is, therefore, important to cultivate the proper species, and if this is done, there would be a large demand for this oil in the country. Extensive trials in the cultivation of *Eucalyptus* were carried out in various places in Northern India, i.e. Saharanpur, Lucknow, Kulu, Kangra valley, Chamba and Ranikhet<sup>6</sup>. As a result of these trials the places most suited for the cultivation of *E. globulus* in Northern India have been found to be Ranikhet, Kangra, Kulu and Chamba, and economic propagation of the plant can be undertaken in these localities.

16. *Glycyrrhiza glabra* Linn. (Liquorice) — Liquorice is cultivated in Italy, France, Germany, China, Siberia, Asia Minor and Turkistan. It grows in the Andamans and other places but the main bulk of the drug is imported into India from the Persian Gulf, Asia Minor and Siberia.

Preparations of liquorice are popular in Western medicine as a mild laxative and in the treatment of cough. It is used as a basis for throat lozenges and for concealing the acrid taste of many nauseating medicines. It is also used in the indigenous medicine.

The plant is easy to grow in river valleys in a warm climate and requires a deep and moderately rich loamy soil. Experimental cultivation was undertaken in the forest nursery at Baramulla at an altitude of 5,500' above sea level in Kashmir and also at the *Indian Institute of Agricultural Research*, New Delhi with success. It will grow well in the temperate regions of the Himalayas, e.g. Himachal Pradesh and in the hill districts of South India.

17. *Hyoscyamus niger* Linn. (Henbane) — It is a well-known medicinal plant which is extensively used for its sedative and other properties. It grows wild at an altitude of 5,000' to 9,000' in the temperate Himalayas. The supply of the drug from the wild plants is insufficient and irregular, and the need for its cultivation has been recognized since 1839. It was successfully grown at the *Royal Botanical Gardens*, Calcutta, Lyallpur, Saharanpur, Agra, Ajmer and Bombay, but the alkaloidal content was found to be low and the drug did not come up to the standard required by the *British Pharmacopoeia*; the cultivation of the plant thus dwindled down. The warm climate

of Saharanpur and other places was entirely different from that which the plants enjoy in a state of nature<sup>8</sup>.

*Hyoscyamus* grows wild throughout the Kashmir valley. Its experimental cultivation was started at suitable places at Yarikah and Drang forest nurseries in Kashmir, 7,000' above sea level. An analysis of the *hyoscyamus* leaves collected from the wild and cultivated plants from the Kashmir valley showed that they contained 0.071 and 0.079 per cent total alkaloids as compared to 0.035 per cent contained in leaves of cultivated plants from Saharanpur<sup>7</sup>.

The results of artificial propagation in Kashmir are encouraging and the Forest Department of Kashmir is planning to extend the cultivation of the drug at suitable altitudes. Attempts to cultivate the plant in other places in the Himalayas such as Kulu Valley and Simla Hills need attention.

*H. muticus* Linn. — The leaves of *Hyoscyamus muticus* Linn. contain a higher percentage of total alkaloids than *H. niger*. The plant is not indigenous to India but can be successfully propagated under the climatic conditions suitable for *H. niger*. In fact, successful experimental cultivation of *H. muticus* has been reported from various places, i.e. Saharanpur, Lyallpur. Seeds of *H. muticus* have been tried in the nurseries of the Forest Department of Kashmir with success.

18. *Hyssopus officinalis* Linn. — *Hyssopus officinalis* is indigenous to countries bordering the Mediterranean Sea and also in Central and Eastern Europe. In India the plant grows wild in the Western Himalayas at altitudes ranging from 8,000' to 10,000' from Kashmir to Kumaon. The leaves of the plant are used for flavouring purposes and it is said to be an ingredient of some of the French liquors. The collection of the plant from natural occurrences is not easy, and it would be worthwhile cultivating the plant in suitable places. The plant has been successfully raised in the forest nursery at Baramulla at 5,500'. The fresh and dry herbs collected from the nursery, on steam distillation, gave the following results<sup>4</sup>.

	Local product		Foreign product	
	Fresh	Dry	Fresh	Dry
Oil yield, %	0.36	0.7	0.07-0.39	0.3-0.9
Sp. gr. of oil, 15°C.	0.9375		0.9270-0.9450	
Ref. index of oil, 20°C.	1.4778		1.4730-1.4860	

The cultivation of the plant can be extended in Kashmir and other regions of the temperate Himalayas such as Himachal Pradesh, Kulu, Kangra valley and other places with altitudes of 4,000' to 6,000'.

19. *Ipomoea purga* Hayne — It is a climbing plant indigenous to the eastern slopes of Mexican Andes. The dried tubers of the plant are known as *jalap* and are extensively used as a purgative in medicine.

It has been observed that the resinous substance obtained from the root of *I. turpe-thum* forms an excellent substitute for the official drug. The plant grows throughout India up to an altitude of 3,000'. The local supply is limited and large quantities of *jalap* are imported from abroad.

The cultivation of the plant was tried in Bengal and the Nilgiris as long ago as 1870. Though the experimental cultivation was not successful in Bengal, it gave very good results in the Nilgiris. Since then it has been cultivated on a commercial scale in Ootacamund and other parts of the Madras Presidency by Government and private agencies. Locally grown *jalap* tubers when extracted after 4-5 years weighed from 5 to 10 lb. and on analysis were found to be as rich in the purgative principles as the best imported tubers from America.

There is a great demand for this drug in India, and its further propagation can be taken up in areas where potatoes flourish. It has been observed that conditions necessary for potato cultivation are equally suitable for *jalap* cultivation. The cultivation of this plant could be tried in the Eastern Himalayas at an altitude of 3,000' to 6,000' in Himachal Pradesh where potato is cultivated on an extensive scale and forms the major cash crop.

20. *Lavandula officinalis* Chaix (Lavender) — The plant is a native of South Europe and grows on the shores of the Mediterranean Sea extending into Western Africa. It is extensively cultivated in England and U.S.A. Lavender oil is a constituent of a large number of pharmaceutical preparations and is extensively used in perfumery. It is not indigenous to India and the entire quantity of about 1288 gallons valued at more than a lakh of rupees is imported from abroad annually.

Experimental cultivation of the plant in the Government Botanical Gardens in the Nilgiris has not given very promising results. It has been successfully grown in the forest nurseries at Baramulla and Chattannar in

Kashmir valley at altitudes of 5,500' above sea level.

The analytical data of the oil obtained from the Kashmir plants are comparable to those of the English oil :

	Local grown	English
Yield of oil, %	2.4 ( dry flowers )	0.8-1.7 (fresh flowers )
Sp. gr., 15°C.	0.919	0.882-0.90
% of ester	24.8	7-14

Large-scale cultivation of the plant such as is being planned by the Forest Department in Kashmir will be a commercial success. The plant should also do well in the Northern Himalayas, e.g. Simla Hills. There is a large demand for this oil in the soap industry.

21. *Lobelia inflata* Linn. (Lobelia) — *Lobelia* consists of the dried aerial parts of *Lobelia inflata* which is an erect herb indigenous to and also cultivated in the Eastern States of America. It is used as an expectorant and in the treatment of chronic bronchitis.

The plant does not grow in India but an allied species *L. nicotianifolia* Heyne grows in a state of nature in South India and Bombay and has been used as a substitute for *L. inflata*. Samples of *Lobelia nicotianifolia* collected in October and November from various parts in India, i.e. Poona, Madras, Tellicherry, etc., show that the Indian drug is even better than the official variety in so far as active principles are concerned. The lobeline content of the drug is 4 times as high (1.8 per cent) as that of an authentic sample of *L. inflata* obtained from New York. The lobeline content of the drug collected in the rainy season is low. Properly collected specimens should consist of flowering tops, slender stems and leaves<sup>16</sup>.

Clinical trials of an ethereal tincture of *L. nicotianifolia* prepared according to the B.P. method showed it to be as effective as the official preparation.

There is room for the systematic cultivation of *L. nicotianifolia* in this country, and steps should be taken to introduce *L. inflata* which may well be tried in the Nilgiris and Travancore hills at altitudes of 3,000' to 7,000'. The plant thrives well in the Eastern Himalayas, Malabar, in the Ghat regions of Bombay and in Bangalore. There is a fair demand for this drug<sup>8</sup>.

22. *Mentha* species: *Mentha pulegium* (Pennyroyal) — The plant is indigenous to most parts of Europe including the United Kingdom, Chile, and many other places with temperate climate. Pennyroyal oil is used

in considerable quantities in perfumery and soap making. *M. pulegium* is not indigenous to India, but has been successfully cultivated at the forest nursery at Baramulla (Kashmir). The essential oil obtained by steam distillation from the dry leaves of the locally grown plants gave the following result on analysis :

	Local	Mediterranean
Yield of oil, %	2.3	
Sp. gr., 15°C.	0.89	0.93-0.95
Ref. index, 20°C.	1.483	1.483-1.486

The cultivation of this plant can be carried out successfully at altitudes ranging from 4,000' to 6,000'. There is a large demand for this oil from the soap industry.

*Mentha piperita* Linn.—This plant is cultivated on an extensive scale in North America, Germany, France and England. The leaves collected during budding and flowering stages of the plant are used for the distillation of volatile oil and manufacture of menthol. The essential oil obtained by distillation of the flowering tops and leaves of the plant is largely used as a stimulant, anti-spasmodic and as flavouring agent in confectionary. This plant is not indigenous to India but was experimentally cultivated in the Nilgiris in 1881 with some success. Experimental cultivation of the plant has also been carried out in Mysore State and at the *Forest Research Institute*, Dehra Dun, but no analytical data are available. Recently the plants were raised successfully from suckers in the forest nursery at Baramulla at an altitude of 5,500'. The dried flowering tops and leaves of the plants were steam distilled and 0.71 per cent of essential oil was obtained as against 0.7 to 1.5 per cent reported from plants grown in England and Russia<sup>4</sup>. There is considerable scope for the cultivation of this plant in suitable localities in India.

*Mentha arvensis* Linn.—This plant which yields the Japanese mint oil, occurs abundantly in Kashmir in a state of nature. The plant yielded 0.45 per cent essential oil but no crystals of menthol separated out when it was allowed to stand at 0°C. The essential oil as obtained from the wild plant did not come up to the standard laid down in the *British Pharmacopoeia*<sup>4</sup>.

Peppermint oil has a fairly large demand in India and in the foreign markets. India imported about 2,000 gallons of the oil annually during the pre-war period but much larger quantities are now in demand. By systematic cultivation in appropriate places

plants which will yield oil of the required quality can be grown. Trial cultivation is recommended in temperate Himalayan tracts at altitudes of 4,000' to 5,000'.

23. *Olea europaea* Linn.—This is a small tree cultivated in France, Spain and in other countries bordering on the Mediterranean Sea and also in California and South Australia. The ripe fruits of the tree are used for expressing the oil. The plant is not indigenous to India but some species of *Olea* grow in certain parts of this country, and it would be interesting to study if the oil extracted from the fruits of these species come up to the official standards.

Attempts to cultivate *Olea europaea* in many parts of India were made long ago, but met with little success. The trees were raised as early as 1800 in the botanical gardens at Bombay, Calcutta, and at Government gardens in Bangalore. The trees attained normal height at all these places, but did not either bear fruits or the fruits did not mature. Plantations were also started in Kashmir State and fruited well but unfortunately the study was not pursued. The cultivation of this plant should be taken up again in suitable localities in the Himalayas. No experimental work has been so far carried out on grafting the European olive on the wild olive tree of India. This may be tried in Kashmir, Himachal Pradesh, etc. The plant should be manured in the same way as is done in Australia and Europe.

24. *Prunus serotina* Ehrh. (Wild cherry) — This tree is widely distributed over North America particularly in Northern Central States. Its bark is collected in the autumn, preferably from young stems and branches. Preparation from the wild cherry bark are used to relieve cough in phthisis, bronchitis, etc. This plant is not indigenous to India, but several other species of *Prunus* grow wild in Kashmir and the Himalayas and some are cultivated for their fruits. Investigations have shown that the bark from these species cannot be substituted for the official drug<sup>17</sup>.

The official plant may well be introduced for cultivation in Kashmir, Kulu, Simla Hills and such other places where the species of *Prunus* are either growing wild or are cultivated. The demand for the bark is not very large.

25. *Piper cubeba* Linn. f. (Cubebs) — This plant is a native of Java, Sumatra and the Malayan Archipelago. The fruits are commonly known as cubebs and are generally used in the treatment of genito-urinary diseases

and as a condiment. Large quantities of cubebs are imported into India from Singapore.

The plant was grown successfully on an experimental scale in Mysore State. The oil distilled from the cubebs was studied by Rao, Sudborough and Watson<sup>18</sup> with the following results :

Yield of oil, %	11.85
Sp. gravity	0.9167
Optical rotation	-29.9°
Refractive index	1.4894
Saponification value	0.5
Saponification value after acetylation	24.1

The data reveal that it should be possible to grow cubebs of good quality in Mysore. Its cultivation may be encouraged in places with warm and moderately moist climates such as those occurring in parts of Travancore, Bombay and Bengal.

26. *Rhamnus purshiana* DC. (Cascara sagrada) — The dried bark of this plant collected at least one year before use is known as Cascara sagrada. Large quantities of the bark, its preparations and its active principle, emodine, are imported into India. The plant grows in North California, Oregon, Washington, and British Columbia. It is a non-irritating mild laxative.

*Rhamnus purshiana* is not indigenous to India but a number of species of *Rhamnus* grow wild in this country. *R. virgatus* Roxb. is one such species which grows in Kashmir and is used as a purgative in indigenous medicine. The bark of this species was investigated both chemically and biologically in order to see if it could be substituted for the official bark. Although the active principles were present, the bark did not come up to the standard of the *British Pharmacopoeia*. Other species are being investigated to determine if any of them could form a suitable substitute. The cultivation of *R. purshiana* is worth trying in temperate Himalayas and other regions with moderate rainfall, i.e. Kashmir, Bhutan, Garhwal, and Nilgiris at altitudes of 2,000' to 5,000', particularly in areas where the other species of *Rhamnus* are growing in a state of nature.

27. *Rosmarinus officinalis* Linn. (Rosemary) — It is an evergreen shrub indigenous to Southern Europe where it grows abundantly in dry rocky hills in the proximity of the Mediterranean Sea. The flowering tops of the plant are distilled to yield the oil of rosemary. The oil is imported from south of France and the Dalmation Islands. It has carminative properties and is employed

principally as spiritus rosmarinus and in hair lotions. In India the plant has been cultivated as an ornamental plant in gardens in a number of places. Its cultivation may well be tried in the plains with equitable climate, and it is likely to do better in temperate Himalayas with dry to moderately moist climates.

28. *Strophanthus kombe* Oliver — This is an important cardiac tonic drug of the *British Pharmacopoeia*. The plant is a native of the African coast, and India imports large quantities of its preparations every year. At least 5 species of *Strophanthus* are indigenous to the tropical regions of India and the Malayan peninsula, but so far no attempt has been made to find out the strophanthine content of these species in order to see if they might substitute the imported variety. *Strophanthus* cultivation would not be difficult under conditions existing in India, and it has been tried experimentally with some degree of success in the *Royal Botanical Gardens*, Calcutta. An investigation into the possibilities of its cultivation in India would be of interest to drug manufacturers.

Some of the *Strophanthus* species grow in Travancore, Malabar and Khasi areas. *Strophanthus kombe* is likely to do well in the drier regions of Southern India.

29. *Theobroma cacao* Linn. — This tree is a native of tropical America and is cultivated in most tropical countries. The seeds are used to extract the oil of theobroma and for the preparation of cocoa powder and chocolate. The bulk of the seed is imported from the West coast of Africa, Ecuador and Brazil.

*T. cacao* flourishes in a hot and moist climate; the young plants must, however, be shaded and well watered. Though not a native of India, it has long been grown in South India and there appears to be no reason why its cultivation should not be extended in suitable places in South India.

30. *Styrax benzoin* Dryand — Benzoin is the balsamic resin obtained from the incised stem of *Styrax benzoin*. It is extensively used as an antiseptic for cuts and wounds, and as an inhalant in chronic inflammation of mucous membranes especially of the respiratory tract. There is a fairly large demand for this drug in India.

The plant was successfully cultivated in the Government Gardens, Bangalore and will probably do well in South India. There are several other species of *Styrax* which are indigenous to India; some grow in Bhutan, Nepal and Sikkim. Attempts are being



made to procure resins from these species and examine their constituents. Cultivation of *Styrax benzoin* may be experimentally tried in these localities and also in South India where the other species of this plant grow in a state of nature.

31. *Valeriana* — The rhizome and root of *Valeriana officinalis* Linn. collected in the autumn and dried slowly constitute this drug. The herb grows in a state of nature, and it is also cultivated. There are two varieties, *Valeriana officinalis* var. *Mikani* Syme and *V. officinalis* var. *sambucifolia* Mikan. The former variety is found wild on the dry calcareous heaths and pastures of Derbyshire in England. The drug is cultivated in Belgium, Holland and France.

A number of species of valerian such as *V. hardwickii* Wall. and *V. wallichii* DC. grow wild in temperate Himalayas. They grow abundantly in mountainous ranges from Kashmir to Bhutan. The plant *V. officinalis* Linn. is also found in the north of Kashmir at an altitude of 8,000' but it is not so common as the other species.

Indian valerian roots have been recommended for use in the pharmaceutical preparations. Valerian is priced in medicine on account of its essential oil. An average specimen yields 0.5 to 0.9 per cent of the oil but the yield varies according to the locality and the season of collection. The demand for valerian has recently been on the increase as it has been found useful in the treatment of shell shock.

The collection of valerian from inaccessible places where it grows in a state of nature is difficult. The cultivation on scientific lines should be taken up in the temperate Himalayas. The drug of standard quality can be ensured by selection of the proper species, careful collection, curing and storage.

There are other less important medicinal plants which do not grow in India but are annually imported into this country. Among them are: *Physostigma venenosum* Balfour which yields physostigmin salicylate of *British Pharmacopoeia*; *Pilocarpus microphyllus* Stapf. which is the source of pilocarpin nitrate; *Myroxylon toluifera* H.B. & K. and *M. pereirae* (Royle) Klotzsch which are the sources of balsam of tolu and balsam of Peru respectively and many others.

A planned programme for the cultivation of drug plants should be taken up by a Government sponsored agency such as the *Bureau of Plant Industry*. If this work is taken up in a systematic manner, India will not only become self-sufficient so far as supply of crude drugs is concerned, but she will also be able to produce these drugs in sufficient quantities for export.

We are grateful to Mr. S. C. Sen, Quinologist to the Government of India, for the valuable information on Cinchona and Ipecacuanha; to Mr. M. K. Thomas, Mycologist, for the valuable information on Ergot production in Madras.

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# REVIEWS

**Currents in Aerials and High Frequency Networks**, by F. B. Pidduck (Oxford University Press, Bombay), 1946, pp. iii+97.

THE BOOK AIMS AT GIVING A BRIEF BUT generalized survey of the behaviour of electrical systems at radio frequencies. It is in essence an extension of Murray's theory and carries the investigation into the field of jointed networks and several other allied circuits.

It opens with an introduction to the subject starting from Maxwell's generalized electro-magnetic equations, and is followed by a summary of the theories of Pocklington and Murray. The fundamental E.M.F. equation for a conductor in a system of  $(\tau+1)$  conductors with arbitrary voltages applied to them is found to be

$$29.98 \sum_{n=1}^{\infty} \sum_{v=0}^{\tau} c_n^v \int_{-l^v}^{+l^v} \left[ \frac{\partial^2 \Theta}{\partial p \partial q} - k^2 \cos \gamma \Theta \right]$$

$$I_n^v(q) dq + (1+i) R^\mu \sum_{n=1}^{\infty} c_n^\mu I_n^\mu(p) - E^\mu(p) = 0$$

This leads to  $(\tau+1)$  infinite sets of equations, one set for each aerial, to solve the coefficients

$c_m^\mu$  in the form:

$$\sum_{n=1}^{\infty} \sum_{v=0}^{\tau} Z_{mn}^{\mu v} c_n^v + (1+i) R^\mu c_m^\mu = U_m^\mu \quad (m=1 \text{ to } \infty)$$

$$\text{where } Z_{mn}^{\mu v} = 29.98 \int_{-l^\mu}^{+l^\mu} \int_{-l^v}^{+l^v} \left[ \frac{\partial^2 \Theta}{\partial p \partial q} - k^2 \cos \gamma \Theta \right]$$

$$I_m^{\mu*}(p) I_n^v(q) dp dq$$

$$\text{and } U_m^\mu = \int_{-l^\mu}^{+l^\mu} E^\mu(p) I_m^{\mu*}(p) dp.$$

$Z_{mn}^{\mu v}$  is one of the mutual impedances of the  $\mu^{\text{th}}$  and  $v^{\text{th}}$  aerials.

These expressions form the basis for the solution of all the problems considered in the book. The case of a single wire is treated by considering the charge on its surface to be equivalent to two parallel line charges, one along the axis and the other along the length. A number of systems are analysed including skew aerials, parallel aerials, loaded aerials, aerials above the ground, Lecher wires, loops, etc. The last article of the book contains a theory of diffraction derived from the above view-point. A very useful list of mathematical tables at the end gives values of the exponential integrals used in the body of the book for various calculations.

From the mathematician's view, the book is a well-written monograph on high frequency phenomena as far as it goes. The style is somewhat terse but most of the calculations are neatly made and detailed work is not included. A few of the minor gaps in the derivations are left to the reader to fill in. This detracts little from the value of the book.

It is when we come to the practical engineer's needs, that the book loses some of its utility. An average engineer will find the book difficult to follow. Many of the calculations and formulae have not been adequately explained physically. We have the case, for example, of the

fundamental expression for  $Z_{mn}^{\mu v}$ . The definitions of  $\mu$ ,  $v$ ,  $m$  and  $n$  would lead us to define this expression as the mutual impedance between the  $m^{\text{th}}$  harmonic of the  $\mu^{\text{th}}$  aerial and the  $n^{\text{th}}$  harmonic of the  $v^{\text{th}}$  aerial. This is in itself obscure, but when we enter into the domain of  $Z_{m,-n}^{\mu v}$ ,  $Z_{m,n}^{\mu v}$  etc., the impedances lose all physical reality and become mere mathematical symbols.

The book does not offer much help in the solution of the daily problems of the engineer. Only special cases are treated and they are of limited scope; too much insistence has been placed on resonant lengths. The behaviour of systems at non-resonant lengths, the variation, for example, of impedance and response of aerials as the frequency is altered, is not to be found in

the book. One or two interesting points are: (a) the determination of the physical length of the resonant dipole and how this depends on the thickness of the conductor; (b) the optimum length across which an untuned feeder should be attached to drive a half-wave aerial for maximum transfer of power. The current in a receiving resonant dipole has been shown to contain third and fifth harmonic components when it is excited by a wave of the fundamental frequency. It is, however, not understood how, in a linear network, third and fifth harmonics can be generated when the exciting voltage does not contain any of them. It has not been explained whether this would apply to a transmitting antenna too.

The printing and other features of the book leave nothing to be desired. There is only one error that is noticeable, — the summation in equation (16) p. 11 should be from  $v=0$  to  $\tau$  and not from  $v=1$  to  $\tau$ . On the whole, the book is a useful and concise mathematical treatment of the limited problems it seeks to solve.

N. L. S. & J. P.

**Chemical Engineering Economics**, by Chaplin Tyler (McGraw-Hill Book Co. Inc., New York), 1948, 3rd edition, pp. ix+321. Price \$ 4.00.

THE BOOK DEALS WITH THE APPLICATION OF economic aspects of chemical engineering to the development of chemical industries. While a sound knowledge of the principles of chemical engineering is necessary for the proper design and operation of individual plant units, for the successful formulation and development of an integrated industry comprising a number of units, it is necessary to consider also other diverse aspects which are normally outside the scope of chemical engineering and to supplement such technical knowledge with additional study of a number of allied subjects. Full appreciation of the basic principles of all such subjects is required to build up sound practical chemical engineering and the author of this book has done a great service to the profession by publishing this book. In the present edition the author has laid particular emphasis on a number of economic aspects which had not been dealt with by him in the earlier editions, and the book has been almost completely re-written.

Although the outlook of the author is mainly American and conclusions are drawn from

experience of operations on scales very much larger than those in vogue in our country, nevertheless the information is extremely stimulating and should considerably help our industrial chemists and chemical engineers in appreciating all the basic factors required in the planning and organization of new chemical ventures.

In the chapter on "Research and Development" interesting figures have been given in regard to the proportion of expenditure for different stages in the formulation of new projects and they should be eye-openers to many of our industrialists; for, there are instances when research and development expenses may comprise 25 to 60 per cent of the total capital needed for the development of a chemical project, although normally even in the case of big schemes it works out to 8 to 10 per cent. Importance of continuous research and orderly development has also been correctly emphasized.

The chapter on "Plant Location" is highly informative, although we have to differ from the author in regard to some minor points. For instance, he mentions on page 52: "sometimes cost of available skilled labour is so high that a manufacturer can afford to train unskilled workers, thus meeting his needs at lower costs". While this may be true for a short period till the unskilled workers get trained up and realize the extent of their contribution to the success of the industry, immediately after this stage is reached, the unskilled workers trained at the cost of the factory will not be less discontent than the directly recruited skilled labour. We are sure that this is not merely our experience in India but true all over the world.

The data of process equipment costs given in the book are up to date and should enable us to evaluate the cost of equipment under present-day conditions fairly accurately.

The chapter on "Plant Design" is a brief review only of the economical aspects of design, and for detailed technical information one has to look for special treatises.

The treatment of "Heat and Power" is extremely interesting and gives a general insight into the importance this aspect plays in the proper organization of a factory.

The later chapters on plant operation and control, cost accounting, market development, research and management are those which we recommend to be read by every technical and business man in our country who has anything to do with the manufacture

and sale of chemicals. They deal with aspects which so far are very little appreciated even by some responsible persons who are today planning the development of chemical industries in India, and a proper consideration of the contents of these chapters will go a long way to helping the development of the Indian chemical industry on rational lines.

The estimates of cost of equipment and cost of production so often given by some research workers in India are extremely misleading and we earnestly request them to go through this valuable book minutely.

There are a few minor printing errors which, however, do not lessen the value of the book in any way.

We recommend this book for the intensive study of not only industrial chemists and chemical engineers, but also by business executives and research workers who have anything to do with chemical industry.

A. N. RAO

**Modern Colloids**, by Robert B. Dean (D. Van Nostrand Company Inc., New York; Macmillan & Co. Ltd., London), 1948, pp. xi+303. Price \$3.75 or 21s. net.

THE RAPIDLY INCREASING VOLUME OF DIVERSE data and original papers characteristic of the recent developments in colloid behaviour has emphasized the need for a general review of the fundamentals of the subject with its recent trends of growth and applications. Professor Dean's "Modern Colloids" is, from this point, a welcome addition to colloid literature. The author presents a coherent and succinct review of the important work on such varied topics as adsorption, high polymers, colloidal ions, foams suspensions and emulsions. Special emphasis has been laid on the applied aspects of the phenomena. The chapters on adsorption, ionic adsorption, and colloidal ions are particularly informative and represent about the latest position in the subject. Chapters on high polymers, plastics, resins and rubber constitute a specially attractive feature of this book, of marked interest alike to research workers and students of general science. One would have, however, welcomed more information on the physico-chemical aspects of these topics.

The book as a whole makes very interesting reading. The numerous illustrations and particularly the molecular models greatly enhance the readability of the material.

Workers in physical chemistry in general, and in colloid chemistry in particular, will be grateful to Professor Dean for providing an up-to-date account of the physics and chemistry of colloids.

S. S. JOSHI

**Fundamentals of Vibration Study**, by R. G. Manley (Chapman & Hall Ltd., London), 1948, 2nd edition, revised, pp. xvi+156. Price 15s. net.

THE FIRST EDITION OF THIS BOOK WAS reviewed in this JOURNAL in its issue of June 1944. In the second edition no fresh matter has been added, because the fundamentals of various kinds of vibration systems had been clearly explained in the first edition. The introduction of "Introductory" and "Summary" sections in each chapter are helpful additions for the study of the subject-matter. Two further appendices giving some applications of the theory to the solution of the simpler practical problems and tables of some of the quantities commonly used in vibration calculations have, no doubt, enhanced the value of this edition.

The book will prove a valuable addition to the technical library of an engineer.

K. B. K. R.

**Soil Erosion — Its Prevention and Control** (Superintendent, Government Press, Madras), 1948, pp. xii+184. Price Rs. 6.

THIS IS A TEXT-BOOK ON SOIL EROSION published by the Government of Madras for the use of departmental officers and for teaching the subject in the agricultural and forest colleges. It has been compiled by a committee consisting of Heads of the Departments of Irrigation, Agriculture, Forest and Public Health, appointed by the Government of Madras.

Erosion has been taking a heavy toll of the soil every year. The extent of its ravages can be judged from the vast tracts of lands that have gone out of cultivation and others whose potential fertility has been reduced to a very low level. The continued existence and prosperity of the country depends on its ability to maintain its productive agricultural land. The control and prevention of soil erosion is, therefore, a subject of great national importance. The present compilation by experts on different aspects of soil conservation will go a long way to meet the demand for a good reference book for workers

in the field of soil conservation under Indian conditions as well as for a good text-book on soil erosion for use in agricultural and forest colleges.

The book contains 18 chapters, 2 appendices, bibliography, glossary, index, and 158 illustrations.

The introductory chapter of the book deals with factors of soil formation, with a general historical account of soil erosion, and describes the problem of erosion in India in general and in Madras in particular. Chapter II deals with the causes and results of soil erosion with interesting illustrations and examples from India and the United States of America. Chapter III deals with the chief causes of accelerated erosion in agricultural soils, such as intensity of rainfall on the catchment, size and shape of the catchment, slope of the land, vegetation, etc., and the experimental technique used for the determination of run off and soil losses. The next four chapters deal with the mechanical methods of erosion control such as contour bunding, contour trenching, terracing and contour ridging. Chapters VIII and IX deal with conservation of grass land and shifting cultivation on hill slopes. Chapter X deals with the biological methods of control of erosion such as regulated forestry, regulated grazing, cover cropping, mixed farming, crop rotations, strip cropping, etc., and the experimental results at the Sholapur and Bijapur farms in Bombay, and Hagari farm in Madras are enumerated. Chapter XI, XII and XIII deal with control of gullies, check dams and permanent soil-saving dams respectively. The material has been prepared so that it may be used by individuals having a limited degree of engineering training and experience. Chapter XIV deals with the special use of vegetation in gully stabilization. Subsequent chapters deal with control of stream and river bank erosion, control of wind erosion, control of floods, and erosion in relation to the spread of malaria. The book ends with a map showing annual average rainfall in India and average monthly and annual rainfall of the Madras Presidency.

The usefulness of the book has been enhanced by the inclusion of good illustrative photographs. The get-up and print are attractive.

A chapter might have been devoted to soil conservation survey and land use classification which are very necessary before planning for maximum safe land use and for adopting wide-scale anti-erosion measures.

This suggestion may be considered in revising the book for the second edition. Comparatively greater emphasis has been laid on the engineering aspects of controlling soil erosion. It is suggested that the biological methods of controlling soil erosion may be described more fully in the next edition.

The book is useful and a copy should be kept in the bookshelf of every worker engaged in soil conservation and general agricultural development.

R. J. KALAMKAR

**Kuchha Pucca Houses**, by Dr. D. R. Dhingra & Shri S. N. Ghatak (Harcourt Butler Technological Institute, Kanpur), 1948, pp. 21+9+iii.

THIS BOOKLET PUBLISHED BY THE *Harcourt Butler Technological Institute*, Kanpur, gives an account of the work initiated by Pt. Keshao Deo Malavia, Development Minister, United Provinces, for developing a suitable stabilized mud plaster for the construction of low-cost houses for homeless millions.

The chief ingredients of this plaster are pond clay containing about 55 per cent silica and 24 per cent sesquioxides, with a plasticity index of 25, mixed with urine-earth, molasses and fibre waste. The mass is allowed to ferment, with turnings, for a period of 30 days. The stabilizing property of the mud is attributed to the formation of a plastic mass similar to urea-aldehyde plastics. It is estimated that 100 cu. ft. of the mud would cost about Rs. 50. The booklet contains details for making the mud and the procedure for its use as a plaster. 3 types of experimental houses have been constructed at the *Institute* costing approximately Rs. 5,500, 2,000 and 1,250 using the new plaster. An antiseptic spray containing kerosene, coal-tar, creosote and copper linoleate has been recommended to protect the wood-work from white ants. Details regarding the specifications, cost estimates and plans are given for each type of house.

The booklet also contains an addendum giving the results of further work on the plaster. In addition to the ingredients mentioned above, press mud from sugar factory waste and bagasse are added after fermentation to give a stronger plaster which, according to the authors, can replace the ordinary country tile roof. The process of manufacturing the mud plaster and its use in building construction have been

illustrated by 36 photographs. Those engaged in building trade would find the booklet interesting.

N. K. P.

**One Story of Radar**, by A. P. Rowe (Cambridge University Press), 1948, pp. xii+208. Price 8s. 6d.

THE BOOK DESCRIBES THE GROWTH, DEVELOPMENT and decay of the organization which was mainly responsible for the development of radar in the U.K. Mr. Rowe was with the T.R.E. (The Telecommunications Research Establishment was a secret till the end of the war) from the beginning, and his story is full of interest to both the scientist and the administrator. The author's aim has been to use radar as an example of how scientific work could and should be organized. Much of the credit for the development of radar is due, as Lord Tedder says in his foreword, to men like Rowe and Tizard who created a real team without losing the spark of individual freedom. "They and men like them set a pattern which is not only vital for our future security, but may well, I suggest, be vital for the well-being of the world."

The administrative story of the development of radar from the first experiments of Sir W. Watt in 1935 to the development of devices like I.F.F., Gee, H2S, and "the fantastic Oboe" by a team of physicists almost actively participating in the conduct

of the war, makes very interesting reading. The more so on account of such thought-provoking statements as "Some genius has said, that victory goes to the side that gets most sleep", "The better is the enemy of the good", etc.

In all the advanced countries, including Germany, it was realized that the scientist should be free from all personal worries, even during a war, and be liberally supplied with the withals of research. In Germany, however, "the German Air Force ruled with harder rod the scientists who sought to serve them, and even at the end of the war the German Airborne Radar was a negligible factor". At T.R.E. not only was a method evolved of "building and maintaining a team of the largest number of first rate physicists ever to work together in the country", but due emphasis was laid on the fact that "what most puts a stamp upon a research establishment and gives it colour, is not its special organizational features but the kind of people it possesses at the upper levels of its structure". The *Sunday Soviets*, the democratic organization of the Establishment, and the fact that Mr. Rowe would worry even about the laundry facilities of his staff, contributed much to what was T.R.E. Also the effect of high morale which success brings, was not negligible; decay set in with the end of the era of spectacular achievements.

B. N. SINGH



# Prefabricated Houses for India

ONE of the most far-sighted moves within the Commonwealth in the field of prefabricated building has been made by the Mysore State Government in India.

At the invitation of the authorities there a British firm of engineers, *Structural & Mechanical Development Engineers Ltd.*, of Slough, sent a technician to Mysore to discuss the possibilities of installing plant for the manufacture of houses.

Accompanying the firm's technician was Mr. A. F. Hare, consulting architect, who carried out extensive investigation of local conditions, availability of materials, costing, etc., and prepared designs based on the *S.M.D.* "Alcrete" construction method.

This work was carried out just over a year ago and the designs met with the approval of the Mysore Government. An Indian company was then formed for the production of "Alcrete" houses within the State.

## Help from U.K.

*S.M.D. Ltd.* are supplying all engineering drawings, production details and have designed and laid out the necessary plant and factory for production, including full specifications and details of specialist processes. They are also supplying full architectural details — including the consultant architect's advice throughout production — and any details for variations which may be produced in this factory at a later stage.

The firm are also providing to the Indian company technical personnel to assist firstly in installing the plant and later supervision of production.

The main part of the house will be produced in India, using local materials. The exports from the U.K. will consist of the small amount of aluminium alloy framework and roof sheetings and, in addition, licences and technical services for the production of the house and specialist processes to be paid for on a royalty and fee basis.

When producing houses of this type, it is most essential that each project should be specifically designed for the particular country concerned, in order that the scheme should be applicable in every way to local conditions.

The "Mysore" house has been designed to give a higher standard of insulation than is normally available there at 5 to 10 per cent lower

cost. Its design allows it to be produced and erected by semi-skilled and unskilled local labour.

## Indian Government's Interest

During the investigations in Mysore, the firm's representatives were ably assisted by the State Architect, Dr. O. H. Koenigsberger. This project was then passed to the Government of India for their consideration. The Government of India have a vast programme for the provision of houses throughout the whole of the country, and it was decided that they should co-opt Dr. Koenigsberger from Mysore State and depute him to visit Europe on behalf of the Government of India to investigate methods of prefabricated housing suitable for India and, particularly, the *Alcrete Method of Construction*.

Dr. Koenigsberger visited Europe in June and July last year and carried out an extensive investigation of the various methods and types of construction, and reported to the authorities at New Delhi. Following this report, an agreement has been signed with *S.M.D.* for a 12-year period for the provision of "Alcrete" houses for India.

Six pairs of prototypes have been ordered to be manufactured and exported from the U.K., and it is the intention to commence work immediately on the installation of the first factory in New Delhi. This factory, which will cost approximately £150,000 (Rs. 19.96 lacs), will be equipped with machinery supplied from the U.K. for the manufacture of 5,500 houses per year. The cost of each house will be just under £200 (Rs. 2,662).

The firm will supply all production details and will send to India an engineer to supervise the installation of the plant and the initial production, and also a Cement Technologist to supervise the production of the "Foamed Concrete" wall panels.

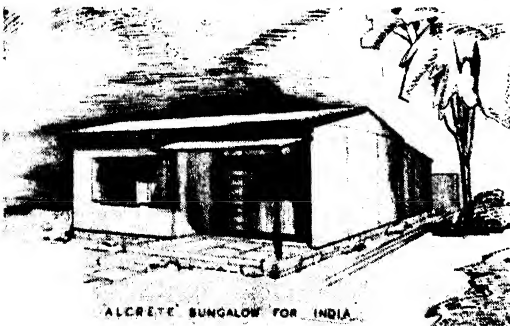
This agreement covers an unstated number of factories, it being the eventual intention to have a number of production units each manufacturing 5,500 houses per year scattered throughout the country. This method of construction, which is basically a method of manufacturing wall panels and roof panels, can be applied to various types of accommodation.

## First Project

The first project is for a very small standard house similar to the standard of housing built in Mysore during the last 8 years, but this system of construction is equally suitable for various sizes of houses — two-storey houses, schools, hospitals, etc., — and it is hoped to combine this system of wall panels with a pre-stressed reinforced concrete frame for multi-storey buildings in areas of high land cost such as Bombay, etc.

The accommodation provided per house is very small compared with Western standards, and comprises two rooms — one 12' 6" × 10' and the other 10' × 10' with a small kitchen and two verandahs, and an external latrine and a bathing place are provided in traditional construction.

The houses, which were designed and planned in Mysore following the investigation of existing



buildings, are naturally very simple and no internal fittings, plumbing, etc., are included. The principle is to provide in the first scheme a basic living unit which is ideally suited to mass production on a large scale at low costs.

The Alcrete wall panels — 3' 2" wide  $\times$  10' high — are made of a light aluminium alloy frame into which is cast a chemically foamed concrete; on to the external face of this is cast a skin of dense concrete and the whole is then cured in autoclaves. The internal wall face is finished in the factory with plaster, and the panel is, therefore, a completed unit requiring erection on site only. This 4" thick wall panel provides a higher standard of insulation than 14" of ordinary brickwork.

The internal partitions are similar in construction to the wall panels; the roof is made of an aluminium alloy girder supporting aluminium alloy corrugated sheeting which is lined with insulating material; the doors and window shutters are also made from aluminium alloy, faced internally with locally produced plywood or asbestos.

#### Fruits of Research

A considerable amount of research has been carried out by *S.M.D. Ltd.* during the past 3 years on the provision of a suitable method of construction for tropical countries — India in particular.

The system of foamed concrete wall panels has been found ideally suitable. This light-weight material provides a high degree of thermal insulation, whilst the thickness of the panels can be varied for individual schemes to provide additional insulation, should this be required; i.e. for India a 4" overall thick panel is provided, but if more stringent conditions were met, this could be increased. The roof also gives a high degree of insulation. This is provided by: (a) a glass silk blanket which is fixed between the aluminium sheet and the plywood inner lining; and (b) the reflectivity of the external aluminium sheet.

It is often not fully appreciated the value which can be obtained by the reflectivity of aluminium sheet when used as a roof covering for hot countries. Aluminium possesses the highest reflectivity value of any of the metals and is second only to silvered glass.

The materials used, aluminium alloy and foamed concrete, are both permanent to a high degree and will withstand all kinds of climatic conditions, and attacks by vermin or insects, etc.

Each country has its own special requirements as to size, shape, amenities, and wants buildings to suit local social conditions, etc. It is, therefore, necessary that each project should be started with a

survey of local conditions and the house then designed specifically to suit the country or district, but fundamentally the same method of manufacture and production would apply.

#### Constructor's Wide Experience

The *S.M.D. Ltd.* provide a typical example of the type of organization in Britain which is prepared to initiate the production of prefabricated houses for interested overseas authorities.

It is primarily a development organization — a subsidiary of *Almin Ltd.* (*Associated Light Metal Industries*) — employing a team of mechanical engineers, structural engineers and research chemists who have been responsible for completely new and original development in many spheres of industry. During the war these technicians designed and supervised the construction of many large chemical and metallurgical plants.

In the post-war period probably their most notable project was the complete initial design, method of construction and development of insulating materials used in the Aluminium House for the U.K. Government.

By far the most highly factory-prefabricated house in the world was produced by a group of 5 large aircraft manufacturers, of which the *Bristol Aeroplane Co.* is the parent company.

The project was sponsored by the Ministry of Supply, *S.M.D.* engineers being retained as consultants to the Ministry for this project and for prefabricated housing in general. The original order placed by the Government for this house was 54,000. Of the various prefabricated single-storey houses in England, the Aluminium type alone is now scheduled as a permanent house and the initial order was increased to 70,000 — the largest contract in the world for one type of housing.

The *S.M.D.* are also manufacturing and exporting aluminium alloy "prefab" storage buildings (*Alframe*) for the Groundnut Scheme in East Africa and various types of transportable buildings. *Alframe* buildings are of 36' clear span to any length of a multiple of 9', and either 9' or 14' 6" in height to the eaves.

These "*Alframe Storage Buildings*" are manufactured in Britain and exported as simple units; being constructed entirely of aluminium alloy they are extremely simple for erection. These buildings are also fully demountable without any new materials or fixings being required. They can be easily erected on a site, used for a period of 6 months, or any number of years, and then demounted, transported to another site, erected and re-used. — (*Courtesy, British Information Services, New Delhi*)



# NOTES & NEWS

## Synthetic Resins in Paper-making

THROUGH THE USE OF A SYNTHETIC resin timbers, which have hitherto found limited application in paper-making, can be employed to yield satisfactory printing paper. The new development, which is the outcome of a comprehensive research programme at the *National Bureau of Standards*, U.S.A., on offset printing papers, does not involve either appreciable increase in costs or any important change in manufacturing methods ( *Franklin Inst.*, 1948, **246**, 252 ).

Deciduous woods, such as maple, birch, beech and poplar, give only short-fibred stock which does not lend itself to hydration by the conventional beating process, and the addition of resins will materially help in increasing the use of short-fibred pulps in making printing paper.

The beating process, employed in pulp-making, provides necessary bonding strength but promotes some of the troublesome behaviour of paper in printing, viz. high expansion, excessive curling, slow oil absorption and show-through of images. The new technique substitutes resin bonds for the gel-like bonds of the hydrated fibres, and gives optimum strength for paper with only a fraction of the beating required to develop comparable strength by beating alone, and provides a superior paper by eliminating all the adverse effects of hydration.

Widely different combinations of commercial wood pulps have been investigated with controlled variation in beating and with and without resin bonding, and in all instances resin-bonded paper was found to be superior with respect to curling, oil absorption, folding endurance, resistance to surface pick and expansivity. Sulphate-cooked aspen pulps and sulphite-cooked birch pulps have produced excellent printing papers with the aid of resin bonding, whereas they can be used only in small amounts as fillers by conventional processing.

Several types of synthetic resins have been employed, with melamine-formaldehyde resins giving the best results. Small amounts, usually less than 3 per cent by weight, are required.

For normal commercial fibre combinations, it was possible to develop as much strength with 2 per cent of resin and no beating as with 9.5 hr. beating without

## New Radio Isotopes

2 IMPORTANT NEW RESEARCH AIDS, hydrogen-3 and helium-3, have been added to the radio isotopes distributed by the *Atomic Energy Commission*, and are available in limited quantities for research purposes.

Hydrogen-3, also called tritium, is a radioactive gas with a half-life period of approximately 12 years. As the only radioactive isotope of hydrogen, it should prove of special value as a tracer in medical, biological and chemical research. Combined with oxygen it forms heavy water, a valuable research tool. Tritium is isolated after the bombardment of a lithium compound by slow neutrons in a nuclear chain-reacting pile, and helium-3 is obtained as the end product of the decay of radioactive tritium.

Helium-3, a stable isotope, is only one-millionth as abundant in nature as ordinary helium, and is expected to provide valuable clues to the still largely unknown properties of the helium nucleus (*Chem. Age*, 1948, **59**, 513).

## Dielectric Lenses for Microwaves

ARTIFICIAL DIELECTRIC LENSES for the development of an antenna capable of passing a very wide band of frequencies have been developed at the *Bell Telephone Laboratories* (*Sci. Prog.*, 1948, **36**, 715). This development will be of great value in the future when radio relay circuits make full use of the broad band widths available.

Lenses made of dielectrics, such as polystyrene, had good focussing properties over very broad bands and were in many ways superior to the other types of antennae for concentrating the beam in the direction of the receiving station. Unfortunately, their great weight for 10' lenses precluded their adoption in place of metallic plate types. The recent development is the construction of what might be termed a *metallic dielectric* which possesses the same characteristics as a true dielectric but which can be made much lighter. Such a metallic dielectric is obtained by

building up a lattice of insulated, conducting, metallic spheres similar to the molecular lattice of a dielectric. For microwaves whose wavelengths are longer than the spacing between the spheres, the artificial dielectric behaves in a similar way to a true dielectric. Provided the lattice spacing requirement is met, the refracting properties of these artificial dielectrics do not vary with wavelength and a lens built up with small conducting elements has broad frequency band characteristics.

The conducting elements can be in the form of spheres or discs made of copper foil to reduce the weight and mounted in light-weight polystyrene foam. For use in radio repeater circuits the lenses are made 10' square, and as only vertically polarized waves are used, the conducting elements are in the form of thin metal strips mounted horizontally. The lengths of strips are varied to give the final assembly a shape similar to that of a convex optical lens, and are mounted between slabs of polystyrene foam. This new type of lens will first be used in the New York to Chicago link.

## New Source of Industrial Alcohol

ETHYL ALCOHOL FROM PETROLEUM refinery processes, instead of the usual fermentation method, is to be obtained from ethylene in a new plant of the *Shell Chemical Corporation* at Houston, Texas (*Chem. Age*, 1948, **59**, 423). The output from this plant will be about 18 million gallons annually. It is estimated that to produce a similar quantity of alcohol by fermentation processes would require 45 million bushels of grain.

The basic raw material for the *Shell* process is ethylene, which is obtained with other olefins in the stream of gases from the catalytic oil-cracking plant at the adjoining refinery of the *Shell Oil Co.* After careful separation and purification, the ethylene is converted by direct hydration into ethyl alcohol. The product is to be sold under the trade name "Neosol" as a proprietary solvent.



resin. Far better strength and resistance to surface picking were obtained with 3 per cent of resin than could be achieved by hydration alone even with the most drastic beating. The addition of 1 to 3 per cent of melamine-formaldehyde resin increases the resistance to surface picking and folding endurance, more than 10 times in some instances. All of the resin-bonded papers showed low expansivity, essential for example, in the printing of maps which may run through the press as many as 15 times.

### Starch Syrup Manufacture

THE PRODUCTION OF SYRUPS FROM starches by the batch acid hydrolysis process is described (*Can. J. Res.*, 1948, **26**, 284). Syrups prepared in semi-pilot plant equipment from wheat, corn, waxy corn and tapioca starches of low protein content were almost identical in taste and appearance. The starches employed had the following composition:

	Ash %	Fat %	Protein %
Wheat	0.26	0.63	0.38
Waxy corn	0.05	0.64	0.32
Potato	0.32	0.12	0.11
Tapioca	0.08	0.27	0.14

It has been found economical to employ starch slurries having a density only slightly below the limit imposed by dilatancy, i.e. 22° to 24°Be. Heavier suspensions result in poor heat transfer of pastes, and charging of the converter becomes difficult. Even with starch of the highest quality, improper technique at the filling stage leads to off-flavour and after-taste.

The method of adding hydrochloric acid is important. With glass-lined equipment it is conveniently introduced with priming water, but with copper converters it is best introduced along with the slurry. A concentration of 0.2 per cent hydrochloric acid based on the dry weight of starch provides adequate acidification for 22° to 24°Be slurries. This method of expressing the acidity ensures uniformity in the sodium chloride content of the final product when slurries of fluctuating starch content are employed.

At the outset of each conversion, sufficient priming water is placed in the empty converter to cover the open-type steam coil; with converters of 2,000 gal. capacity approximately 100 gal. of water are required for this purpose. The water is brought to

### Forthcoming International Congresses — 1949

Date of Conference	Title of Conference	Sponsored by	Place
Feb. 2	7th Pacific Science Conference	Royal Society of New Zealand	Auckland & Christchurch
Feb. 8	International Civil Aviation Organization; Air Line Operating Practices, Operations Division	International Civil Aviation Organization	Montreal
Feb. 22	International Civil Aviation Organization; Airworthiness Division	International Civil Aviation Organization	Montreal
May 16	U.N. Scientific Conference on Conservation & Utilization of Resources	ECOSOC	U.S.A.
May	International Railway Congress	International Railway Congress Assoc., Brussels	Lisbon
June 14	Committee on Science & its Social Relations	ICSU	Paris
June 20	International Conference on Science Abstracting	UNESCO	Paris
July 9	4th Empire Mining & Metallurgical Congress	Empire Council of Mining & Metallurgical Institutions	London & Oxford
July 21	2nd International Congress of Crop Protection	International Union of Chemistry	London
July	Commonwealth & Empire Conference on Tuberculosis	National Assoc. for the Prevention of Tuberculosis	London
July	Congress of Psychotechnics	National Institute of Industrial Psychology	Berne
Aug. 15	12th International Dairy Congress	International Dairy Federation	Stockholm
Aug. 19	1st International Biochemical Congress	Biochemical Society	Cambridge
Aug. & Sept.	Specialist Conference on Plant & Animal Nutrition in Relation to Soil & Climatic Factors	C.S.I.R., Australia	Australia
Sept. 6	15th General Conference	International Union of Chemistry	Amsterdam
Sept. 14	General Assembly; International Council of Scientific Unions	ICSU	Copenhagen
Oct.	5th International Animal Husbandry Conference	...	Paris

boiling by steam injection before the starch slurry is let in. During the filling operation, which must proceed continuously, slurry addition and steam injection are integrated so that boiling of the converter contents proceeds vigorously, with steam escaping freely from the air vent. After the converter has been filled and swept out thoroughly with steam, the pressure is raised to 35 lb./sq. in. The progress of hydrolysis is followed by iodine test until the hydrolysate having a *D.E.* (Dextrose equivalent) value of 45-50 is obtained when they are immediately removed from the converter. A *D.E.* value of 55 is regarded as the upper limit for the acid hydrolysis process because of the off-flavour and crystallization tendency of more completely hydrolysed products. The hydrolysates are neutralized with sodium carbonate which is added with continuous agitation as rapidly as the foaming tendency will permit. Gradual additions are then made with periodic pH measurements, until a pH value of 5.0 to 5.1 is attained.

In industrial practice, fatty impurities are skimmed off, and protein and other impurities flocculated by the addition of Hyflo Super Cel (0.1 to 0.2 per cent on the weight of liquid) and filter-pressing the syrup. Activated carbon (0.2 to 0.5 per cent on liquid weight) is added, heated for 20 min. at 80°C., filter-pressed and evaporated to 30°Be *in vacuo* or at atmospheric pressure. A further 20 min. treatment at 80°C. with 0.5 per cent carbon and 0.1 per cent flocculating agent on 30°Be syrup weight removes the colour developed during the first evaporation. The final filtration at this density must effect complete removal of suspended impurities. In evaporating the heavy syrup *in vacuo* to the final density of 42°-43°Be control density measurements are conveniently taken at the evaporation temperature of 140°F., a correction of 1.0°Be being added to obtain the conventional density value at 100°F.

In decolorizing with bone char, the neutralized hydrolysate is defatted, freed of flocculated proteins by filtration with diatomaceous earth before percolation through gravity filters. After evaporation to 30°Be the syrup is again decolorized with bone char and filtered if necessary before its evaporation to final

consistency. Bone char has been found to be superior to activated carbon with respect to adsorption of copper and iron traces as well as salts in industrial waters. It was also found necessary to extract fresh bone char exhaustively with hydrochloric acid before use, otherwise decolorization is slow and the syrup turns dark during evaporation.

Syrups prepared from starches that were heavily contaminated with protein were consistently objectionable in taste and appearance, and bitterness could not be removed by repeated passage through bone char filters. Nitrogenous impurities cause troublesome frothing in evaporators and affect the usefulness of the syrup in the confectionary and soft drink trades. Considerable haze also develops in finished syrups of high nitrogen content. The haze can be largely corrected by diluting to 25°-30°Be filtering off the flocculated impurities and concentrating. Purification of starch before it enters the converter appears to be the only practical measure that can be taken to provide a satisfactory syrup.

Tap water promoted colour development in the evaporator and inhibited colour removal by activated carbon. Colour and fluorescence development in stored syrups was promoted by protein impurities, light and tap water and was strongly inhibited by sodium bisulphite. Syrups which remained colourless almost indefinitely were prepared from prime quality starches by using distilled water in the process and either acid-extracted activated carbon or bone char as decolorizing agents.

#### Deodorization of Shark Liver Oil

THE DEODORIZATION OF SHARK liver oil under different conditions has been described (*Nature*, 1948, 162, 494). The deodorization has to be effected in such a manner as to retain vitamin A intact. The methods tried are: (1) steam distillation of the oil under normal and reduced pressures; (2) agitation of the oil with fermenting milk and toddy; and (3) selective hydrogenation of the oil in the presence of a nickel catalyst. Steam-treated oils are fairly free from odour when freshly prepared but revert in course of a few days to their original character. Oils deodorized by agitation with fermenting

milk or toddy, have been found to remain bland for several months and show very little change in their potency or chemical constants.

Controlled hydrogenation of fish liver oils using 0.1, 0.25 and 0.4 per cent concentrations of nickel catalyst has given promising results. 0.25 per cent of the catalyst effected fairly complete deodorization of the oil at 120°C. within 30-45 min. The percentage loss of vitamin A by this process was about 7 and stability studies have shown that the keeping quality of the oil improved considerably.

#### Contact Acid from Arsenical Pyrites

THE MODIFICATIONS INSTITUTED in the contact sulphuric acid plant of the *Nitrogen Fertilizers Ltd.*, Flixborough, to deal with arsenical pyrites as raw materials are described (*Chem. & Ind.*, 1948, 67, 595). The ore employed normally contains 0.4-0.5 per cent of arsenical impurity by weight.

On the basis of the factory practice and experience, the following tentative specification of the pyrites to obtain burner house efficiency has been drawn up: size,  $\frac{1}{8}$ " to  $\frac{3}{8}$ "; copper, not more than 0.5 per cent; zinc, not more than 1.7 per cent; arsenic, not more than 0.15 per cent.

The gases from the burners after passing through the electrostatic precipitators still contain appreciable amounts of arsenic and lead. In the 2 wash towers, the normal practice is to use 60 to 70 per cent sulphuric acid in the first and 40 to 50 per cent acid in the second. The impurities remain suspended in the wash acid of the first tower, and settle down as sludge in the cooling towers which have to be periodically desludged. In addition, hard scales are formed in the cooling coils which adversely affect the efficiency of heat transfer. Studies on the solubility of arsenic in sulphuric acid have led to the conclusion that operating the first wash tower with 30 to 40 per cent acid and the second with 10 to 15 per cent acid, the wash tower systems remain clear for several weeks. The weaker acid has a greater capacity to hold arsenic and iron in solution resulting in a cleaner circulation acid and lesser possibility of scale formation in the cooling coils.

The use of weaker acid is attended with certain difficulties so far as its use for ammonium sulphate manufacture is concerned. A greater amount of soluble iron is added to the saturator system requiring increased consumption of expensive phosphoric acid.

### A New Aluminium Alloy

A NEW ECONOMICAL DIE-CASTING aluminium alloy has been developed at the *Federated Metals Division of the American Smelting & Refining Company*, New York. The alloy is made of copper, silicon and aluminium and is said to overcome former problems of drilling, tapping and machining of castings made from other alloys. The new alloy will be known as F-4110, under a nomenclature system developed by the *Division*, which reveals the composition of the alloy. It contains 4 per cent copper, 11 per cent silicon, no magnesium and the balance aluminium. There is less tendency for castings to weld to the die during the casting process. It has higher tensile strength, yield strength and proportional limit and higher hardness. The castings assume a whiter finish after casting and the final cost of casting is lower because the alloy is generally cheaper than the primary 12 per cent silicon alloys (*Chem. Age*, 1948, 59, 356).

### New Process for Pig Iron

A NEW PROCESS FOR THE PRODUCTION of pig iron, stepping up the production of existing furnaces by at least 20 per cent, has been perfected by the *Republican Steel Corporation*, of Cleveland, Ohio. A "pressure blowing" technique is employed which, under certain conditions, is claimed to give an increase of 50 per cent in output. The method has been tested in converted blast furnaces of the company. The furnace is partially sealed by a system of valves to retain the hot gases and the incandescent atmosphere thus resulting builds up a high pressure permitting the production of higher temperatures in a shorter time (*Chem. Age*, 1948, 59, 481).

The cost of converting the furnace is reported to be much less than the cost of erecting a new furnace. Apart from increased production, other advantages cited are increased efficiency in burning coke, more uniform quality of iron, and less flue dust.

### "Solder" Glasses

A GROUP OF VERY SOFT GLASSES have recently been developed at the *B.T.H. Research Laboratories* by means of which 2 pieces of glass can be "soldered together without distortion" (*Sci. Prog.*, 1948, 36, 714). These solder glasses, as they are popularly termed, are lead borate glasses whose resistance to weathering has been improved by the addition of zinc oxide. By comparison with other types of glasses, the temperature at which they become soft is low, usually well below 460°C. A solder glass with a suitable coefficient of expansion has been successfully employed to join the soda-lime-silicate bulb in miniature radio valves to the lead glass base without distorting either.

The solder glasses can also be employed to seal mica to metal or glass, as well as metal to glass. With the aid of this technique, a piece of soda glass tubing has had a mica window sealed on to one end, a glass plate on to the other, and a sheet of nickel-chrome-iron alloy sealed across the middle to form a diaphragm.

### Identification of Formaldehyde-resin Finishes

A METHOD FOR THE IDENTIFICATION of formaldehyde-resin finishes on cellulose fibres and wool is described (*C.T.J.*, 1948, 123, 409). 1 gm. of the material to be tested is extracted by boiling twice for 5 min. with 50 cc. of pure alcohol and rinsing 3 times with 50 cc. of distilled water for each rinse. The cleansed sample is heated with 1 per cent solution of pure hydrochloric acid, the distillate filtered, and 3 cc. of it introduced into a freshly prepared solution of 0.005 gm. pure carbazole in 5 cc. chemically pure sulphuric acid. The presence of formaldehyde is indicated by the development of a blue-green colour in the region of contact between the distillate and the test solution.

The hydrochloric acid extract is tested with 5 per cent solution of ammonium molybdate for resins. A white flocculent precipitate indicates melamine resins or proteins. If this test is positive a further portion of the acid extract is mixed with 1 per cent solution of picric acid when melamine resins produce a pronounced turbidity. Proteins do not cause a turbidity unless they are in a very high concentra-

tion. If the above 2 tests are inconclusive, the extract is treated with a 5 per cent solution of potassium ferrocyanide, when melamine resins alone produce a turbidity; proteins and urea resins are inert.

### Electron Microscopy of Natural Cellulose Fibres

3 NEW DEVELOPMENTS IN THE technique of preparing specimens for electron microscopy,—metallic shadow casting, surface replicas, and electron stains, have been employed for the study of the sub-microscopic structure of natural cellulose fibres (*Ind. Eng. Chem.*, 1948, 40, 1711). The results of this investigation offer additional evidence in support of a fibrillate structure for natural cellulose.

The coarse fibrils commonly observed by optical and electron microscopy are shown by metallic shadow casting to consist of still finer unit fibrils, whose diameter varies from 90 to 400 Å depending on the sample. Although the diameters of these fine fibres vary somewhat in a single sample, greater differences in this respect are observed among fibres of different origins. The diameters of the fibrils in ramie, cotton and wood pulp are found to decrease in the order named. Similarly, the variations in straightness follow the same order. These differences suggest a correlation between the character of the fibrils and the chemical and physical properties of the fibres. Ramie fibrils, for example, are found to be highly crystalline and oriented with marked tensile strength but with low elongation and poor reactivity. The properties of wood-pulp fibres are in some respects opposite to those of ramie, whereas cotton is, in general, intermediate.

### Preservative Treatment for Bamboos

EXPERIMENTS ON THE TREATMENT of green bamboos with inorganic preservatives are described in the *Indian Forest Bulletin*, No. 137 (1947). The species tested were *B. arundinaceae*, *B. nultans*, *B. tulda*, *Melocanna bambusoides*, *D. strictus* and *D. hamiltonii*. The bamboos were normally about 15 ft. long. The preservatives tried were zinc chloride, "Ascu", copper sulphate, boric acid and borax. 3 processes were investigated: (a) the original Boucherie process, where fresh bamboos were allowed to stand in a container of

preservative; (b) the normal Boucherie process, where the preservative solution is held in an old cycle tube attached to the pole and held at a higher level than the crown; and (c) modified Boucherie process which consists in using the basal internode as the reservoir for the solution and standing the bamboo in a vertical position. The preservative is introduced through an incision at the node.

Bamboos treated with a 16 per cent solution of zinc chloride for 5 to 6 days gave satisfactory results by all the 3 methods and provided adequate protection against termites, borers and fungi. Felled and stored bamboos can be treated similarly after they are kept immersed in a log pond for 2 to 3 days.

### Technological Developments in Japan

THE PROGRESS IN THE JAPANESE chemical and textile technology is described in Summation No. 32 of Non-Military Activities in Japan, issued by SCAP (*Chem. Age*, 1948, 59, 486).

Among the developments described are experiments to determine new uses for a synthetic fibre made from polyvinyl alcohol. Polyvinyl acetate is saponified by alcoholic caustic soda and the resulting solution is spun in a bath containing zinc sulphate and Glauber's salt. The fibre is rendered insoluble in the presence of formalin, Glauber's salt and sulphuric acid. The new fibre is claimed to have many advantages over other synthetic fibres. It is resistant to acid, alkali and oil; it is stronger and more resistant to abrasion than ordinary fibres. Because of its exceptional strength it can be used for fish lines and nets.

A process for the production of coke of a quality suitable for large blast furnaces has been developed by the *Fuel Research Laboratory* of the Japanese Ministry of Commerce and Industry. Indigenous coal, which does not yield coke of a sufficiently high calorific value with normal coking processes, is used as the raw material. In the new process, which has already reached the pilot-plant stage, pulverized coal is mixed with an equal amount of creosote oil and heated to 350°C. for 10 hr. From the resulting pitch-like substance, a coke of high calorific value can be made by the usual high temperature process of coke manu-

facture. Both non-caking and caking coals have been successfully employed. The product can be used for blending with light caking coals to supplement the supply of imported coking coal.

To relieve the critical shortage of caustic soda, a direct method of electrolysing sea water is being experimented with at the *Teikoku Rayon Co. Ltd.* 8 small electrolyzers are producing approximately 15 kg. of caustic soda per day. The purity of the product is reported to be 99.8 per cent and it is recovered at 15-20 per cent concentration. Chlorine and bromine are obtained as by-products. The process requires 12,000 kW. for the production of each ton of caustic soda as compared with 3,000 kW. necessary using ordinary electrolytic methods. This is largely offset by the saving in power, fuel and labour required to produce and purify salt.

Of the recent inventions in Japan, the following offer good prospects for industrial development: the use of waste middle oil (*B.P.*, 250°-300°C.) after repeated use in the separation of benzene and heavy oil from coal gas until it possesses no longer any absorbing power, as a basic vehicle for printing ink. The simultaneous manufacture of white phosphorus and phosphate of potash fertilizer has been accomplished by mixing limestone and potassic feldspar or other potassium-containing minerals with phosphate rock, adding a small amount of carbon as a reducing agent. The yield and purity of phosphorus is said to be high.

### A New Mass Spectrometer

A SIMPLIFIED AND FLEXIBLE radio-frequency mass spectrometer has been designed by Dr. W. H. Bennet of the *U.S. National Bureau of Standards* for the detection, separation, identification and measurement of negative atomic ions of heavier metallic elements (*Chem. Age*, 1948, 59, 420). The instrument has a specially developed vacuum tube. The spectrometer not only provides a new means of exploring the little known fundamentals of negative ions, but its special characteristics make it widely applicable for other uses including the mass spectrometry of positive ions and for ionization studies.

This two-stage instrument consists of a multigrad tube in which

an adjustable radio-frequency is applied to 2 grids, while all other electrodes are held at the proper direct current potentials and the ion current is measured at the plate. The limitations of the ordinary mass spectrograph using magnetic deflection are overcome in this instrument as the voltage of the ions can be increased to any required value for which insulation can be provided.

The simplicity and low cost of the radio-frequency mass spectrometer, the *Bureau* holds, should make it attractive not only in those applications in which its special characteristics make it superior to other kinds of mass spectrometer, but also in those laboratories where the expense of other types of equipment is prohibitive.

### A New Glass Pump

AN IMPORTANT DEVELOPMENT IN pump design has been introduced by *Tungstone Products Ltd.* Made of pyrex-borosilicate glass, it makes practicable the safe conveying of all acids and alkalis with the exception of concentrated hydrofluoric acid and glacial phosphoric acid. The use of glass is made possible by the absence in the pump of all centrifugal and reciprocating stresses. The pump has the additional advantages of visibility cleanliness, and a capacity for withstanding temperatures up to 200°C. It can be flushed with steam or hot acid solutions. With a capacity (4' inlet head) of 300 gal. per hr., up to 50 lb. per sq. in. delivery pressure, and requiring 1.5 to 4.5 cu. ft. of free air per min., the pump weighs only 54 lb. and measures 13"×13" by 30"—overall height.

### Antibiotics in Onion & Disease Resistance

THE RELATION OF ANTIBIOTIC substances in the scales of different varieties of onions to disease resistance characteristics of the bulbs has been recently investigated on a field scale (*J. Agr. Res.*, 1948, 77, 115). White, red, yellow and brown varieties have been investigated for their resistance to infection by *Colletotrichum circinans*, *Aspergillus niger* and *Botrytis alli* which cause the diseases popularly known as black smudge, black mould and neck rot respectively. The investigations were conducted on crops during 4 seasons.

The investigations revealed that while coloured varieties were in

general more resistant to smudge, there were some notable and rather consistent exceptions. Strongly pungent varieties in red, yellow and white groups tended in some seasons to have significantly lower smudge incidence than mildly pungent varieties. In *F*<sub>2</sub> progenies from crosses between white strong × yellow mild and between white strong × yellow strong, the heterozygous class from the latter cross had a consistently lower smudge index. These facts indicated that pungency had an effect on the varietal resistance to smudge, but it did not explain some outstanding cases where a coloured strong variety had a smudge index not significantly different from those of mild varieties.

The character of the outer scales of varieties varied greatly in number and in tendency to split so as to expose the fleshy scale. The poor outer scale character is a factor intimately related to high smudge incidence. The outer scale character is regarded as the most important condition modifying the smudge resistance of coloured varieties which is due primarily to the phenolic antibiotics in the outer scales.

During one season when neck rot was severe, a distinct correlation was found to exist between pungency and disease resistance. There is also a positive correlation between resistance and toxicity of the volatile and non-volatile antibiotics in fleshy scales. In one season the deeply coloured Australian brown showed high incidence of black mould. The extract of dry outer coloured scales was distinctly stimulative to germination and growth of *A. niger*. It appears that the same phenolic substances which are the major factors for resistance to smudge and neck rot may be the ones which contribute to black mould susceptibility in coloured varieties.

### Australian Cotton Hybrids

COTTON HYBRIDS GROWN AT THE *Bijola Regional Experimental Station*, Queensland, have shown promise of high yielding quality, high ginning percentage and high *jassid* resistance, together with large bolls. Some of them yielded 2,240 lb. of cotton to the acre under irrigation compared with 1,700 lb. from commercial varieties now in use. The staple length of the hybrids approximates to that of the *Miller* variety.

### Chemical Research in Britain, 1938-46

THE REPORT OF THE *Chemistry Research Board*, D.S.I.R., London, for the period 1938-46, published recently, reviews the work of the *Chemical Research Laboratory*, Teddington, during the war period and the period since the cessation of hostilities.

The work of the *Corrosion Section* acquired additional importance during the war, particularly as regards naval craft and armament. The *CRL* rotor test, designed primarily for fundamental studies of immersed corrosion, gave valuable service in assessing the relative values of a variety of protective coatings. Research into corrosion inhibitors has led to the discovery of the inhibitive properties of sodium benzoate, both as an addition to aqueous solutions and as an impregnant into wrapping materials.

Considerable effort was devoted to the alleviation of war-time shortages by the development of processes for the production of materials normally imported. An example of this is the work on carbon black. It has been shown, that suitable carbon blacks can be prepared from indigenous materials, particularly coal-tar products. Highly successful foam fire extinguishers were developed for fighting petroleum fires. As an insurance against protein shortage during the war, the development of a food yeast from *Torula utilis* was given considerable attention and pilot plant studies were completed. On the basis of this development a full-scale plant was erected in Jamaica for the production of food yeast. A shortage of *derris* for insecticidal purposes led the *Laboratory* to collaborate with the *Agricultural Research Council* in the development of effective materials for the control of animal parasites, and a satisfactory DDT emulsion was finally formulated for the control of blow-fly.

A few selected topics have been proposed for investigation during the post-war period. They include: (1) isolation and identification of tar products; development of technical methods for the isolation of tar components and the determination of physical and physico-chemical constants of hydrocarbons; (2) studies on corrosion of metals under immersed and atmospheric conditions, and also of soil corrosion and development of accelerated

tests and of new surface treatments for protection against corrosion; (3) a study of the processes by which ore deposition occurs in nature and of the methods of recovery of semi-rare metals particularly in low-grade materials; (4) development of new high polymers and a study of the relation between chemical structure and physical and mechanical properties of plastics; (5) preparation of organic intermediates capable of polymerization and a study of organic reactions under pressure; (6) microbiological investigations relating to the transformation of carbohydrates and the production of chemical substances by micro-organisms. Study of the reactions of sulphur-reducing and sulphur-oxidizing organisms. The studies may be limited to microbiological problems connected with corrosion.

The work on these problems made substantial progress during 1946. Research on more fundamental aspects of metallic corrosion has been resumed. The rotor test has been employed in the study of immersed corrosion. Work on electrode potential measurement as a means of detecting breakdown of painted surfaces was started. A method of stripping oxide films on copper, iron and other metals, originally discovered by Dr. Ulick R. Evans, has been improved by the exclusion of water and oxygen to give quantitative removal of the films so that its thickness and composition can be determined. Recently the invisible oxide film responsible for the protection of stainless steel has been separated and analysed. An enrichment of chromium in the film has been established. Accelerated corrosion tests are being explored, using conditions which reveal the known superior corrosion resistance of phosphated and painted surfaces as compared with painted but untreated surfaces. Strains of anaerobic bacteria (*V. desulphuricans*) are being intensively studied in relation to the general mechanism of anaerobic corrosion. New methods of extracting germanium and gallium from low-grade sources have been worked out. A preliminary investigation on the separation of isotopes by base exchange methods have been commenced.

A study of the chemistry of functional groups of high polymers has been initiated with the

object of disclosing new techniques for the elucidation of chemical structure and for the preparation of special polymers. Considerable progress has been made towards a clearer understanding of the relationships existing between the properties of reinforced plastics and those of other components. An investigation is being made on the influence of fat and wax, as impurities, on the adhesion between cotton fibre and phenolic resin. The commercial development of synthetic adhesives for wood has enabled high joint strength to be maintained under most severe climatic conditions but these glues are expensive and require great care in handling. A neutral cold-setting glue which overcomes the latter disadvantage has been developed from resorcinol and formaldehyde. Certain special applications of ion-exchange resins are being investigated.

The work now initiated on coal-tar includes a study of newer separation processes, the preparation of tar constituents in the purest attainable state and the determination of physical and physico-chemical constants with high accuracy. As an example of recent work on the isolation of pure components is the recovery from coal-tar base fraction of  $\beta$ -picoline. Among its 3 constituents, 2:6-lutidine,  $\gamma$ -picoline and  $\beta$ -picoline, the last compound has a current importance as an intermediate for the production of nicotinic acid. The separation of the 3 individual bases was effected by azeotropic fractional distillation, using acetic acid as azeotropic former. A method for recovering the acid for reuse has been developed. Attention is being paid to find large-scale industrial uses for higher aromatic hydrocarbons of coal-tar. Acenaphthene, present in coke oven tar to the extent of 1 per cent, and readily extractable in pure form, shows promise of yielding industrially valuable plastics with high softening point and good electrical properties.

The high pressure equipment in the *Chemical Research Laboratory* has been used in 2 important lines of investigation, one leading to an improved process for converting phenols into corresponding aromatic amines, and the other for producing higher fatty alcohols from the corresponding acids.

At the request of the *Board of Trade* an investigation on the methods of preventing the caking

of mixed fertilizers has been commenced.

Other matters receiving attention are: development and production of acetylene and its utilization as an intermediate product in chemical industry; field trials with phenanthridium compounds in the control of *Nagana* in Africa.

### Co-ordination of Cartographic Services

THE *United Nations Organization*, early in 1948, approached interested international organizations to submit relevant information and comments upon the Brazilian Resolution on "Co-ordination of Cartographic Services". Accordingly, a meeting of the experts and representatives of a few international organizations was convened during June 2-3, 1948 in Washington under the joint sponsorship of the *Pan American Institute of Geography & History* and the *International Council of Scientific Unions*.

The members of the Conference were satisfied that the term "Cartography" in the Resolution was employed in its fullest sense, i.e. all types of surveys and of maps and their attendant by-products and the graphic portrayal of economic, social and cultural factors.

The three basic ideas contained in the Resolution are: (a) that the Council recommend to the Member Governments the stimulation of the accurate surveying and mapping of their national territories; (b) that the U.N. Secretary-General take appropriate action to further such efforts by specific means including the advancement of the science itself; the preparation of an up-to-date study on modern cartographic methods; and the development of uniform international standards; and (c) that the cartographic services of the United Nations and the specialized agencies be co-ordinated, taking into account the work of the various inter-Governmental and international non-Governmental organizations.

The consensus of the group as to the most practical way to undertake an appropriate study of the Resolution would be the calling of a meeting of experts and representatives of the interested agencies and international organizations.

Attention was drawn to the concrete contributions which existing international organiza-

tions and specialized agencies can make by providing available reports on (a) their achievements to date in the development of a body of uniform international standards, including their proposals for the future, and of (b) their past accomplishments, present programmes and future plans in the field of cartography.

The urgency of an early meeting of experts and also of adequate documentation, therefore, was stressed by the group so as to enable the proposed meeting to come off early i. 1949. The specific interests of various participating organizations were considered, and it was felt that it would be desirable to give an idea of the nature of discussion on the subject which the several organizations might wish to take under advisement. The following draft was submitted by the President of the *International Council of Scientific Unions* to facilitate other organizations in their consideration of action to be taken at the forthcoming meeting:

"The *International Council of Scientific Unions* desires to stress to the Secretary-General of the United Nations its urgent sense of the need for a full realization of the general import of the Resolution on 'Co-ordination of Cartographic Services of Specialized Agencies and International Organizations', particularly, with reference to the scientific and technical aspects of cartography. Besides national and international interests and other scientific and technical interests, such as hydrographic, aerological, geological, surveying and mapping, and similar fields of scientific endeavour, those of the *International Council of Scientific Unions* bear significantly on this subject as evidenced by its autonomous member Unions of Geography, Geodesy, Geophysics, Astronomy, Scientific Radio and Scientific Biology (and eventually Mathematics, now being reorganized).

"At present, there exists duplication of effort and of personnel in these varied and related fields. A considered co-ordination of these overlapping activities must perforce result in the economical use of funds and in advantage to future advance in the cartographic science and its application to human welfare.

"Some indication of the interest of the *International Council of Scientific Unions* in the suggested International Cartographic Orga-

nization was set forth by Professor E. de Martonne in his communication to the U.N. Secretary-General. It was stated that such an organization would help : (1) to expedite the survey of the world and propagate the results of this survey ; (2) to unify cartographical methods and processes ; and (3) to support and promote cartographical works of the type of the National Atlas. The propagation of the progress of cartography in each country would not only be a boon to all the theoretical and research bodies depending upon geographical knowledge, but would also serve as a stimulant for the progress of this knowledge.

" This progress could be achieved by the publication of an international cartographical bibliography, mentioning not only the maps published by national cartographical services, but large- and small-scale maps representing the extent of physical and economic phenomena . . . " Scientific and practical advances in the studies of the earth sciences and phenomena thereof involve cartographic matters of first importance in geodesy, meteorology, seismology, geology, geomagnetism, oceanography, vulcanology and hydrology. Other Unions undoubtedly have similar interests, such as scientific biology, scientific radio, astronomy and mathematics.

As complementary to co-ordination activities in the field of cartography and related sciences, and of cartographic services internationally, the group invited attention to the polar regions as an area of the world in which the co-ordination of cartographic operations would yield immediate and fruitful results.

### Inter-University Relations

THE QUESTION OF INTER-UNIVERSITY relations was discussed at a recent *Congress of Universities of the Commonwealth*. The problems discussed covered both administrative and academic aspects such as, business arrangements between universities as will enable a student or a teacher to move from one university to another without academic loss of standing or financial embarrassment, and to make such an exchange sufficiently attractive from an academic point of view as to research and other facilities. The *Congress* agreed to recommend that the universities of each country should be encouraged to form an agency which should act

as an instrument of communication with universities elsewhere. Further, since the *Congress* meets only every 5 years, it was suggested that, in the years between such meetings, vice-chancellors and senior members of staff should visit each other as frequently and as regularly as possible.

The *Congress* passed several resolutions designed to make the traffic between universities easier and less hazardous. They were : (1) to set up a *Central Office of the Universities Bureau* to announce and advertise vacancies. Such a *Bureau* will arrange for candidates to be interviewed by a panel of experts in that particular field and the successful candidate to be supplied with information relating to the facilities at the university he is to work in ; (2) the question of superannuation benefits to be taken up so that a worker is not made to suffer financial loss if he goes to other universities ; a *Committee* was set up to enquire into this question ; (3) foundations and other bodies to be approached for financial help to meet travelling expenses of scholars (*Nature*, 1948, 162, 585).

### Industrial Research in France

THE SETTING UP OF INDUSTRIAL research centres to serve the needs of industries is facilitated by a recent legislation enacted in France. Certain industries had, prior to this legislation, set up representative research bodies, of which the *Centre Technique des Industries de la Fonderie* is an example. This body derives its income from a levy consisting of obligatory contributions from firms in all branches of the founding industry, ferrous and non-ferrous, and supplemented by payments for specific investigations undertaken at the request of firms. The aim of the present legislation is to define the status, and regularize the methods of inaugurating new industrial research bodies without materially affecting the position of those already in existence.

Under the new Act, research centres may be created to serve any industry, provided that a sufficient degree of agreement and support is forthcoming from appropriate trade associations and organizations representing the industry. The approval of the Minister for Industry and Commerce must then be secured. The aims of industrial research centres

are to further technical progress, to take part in increasing production, and to ensure the quality of products of the industry with which they are associated. These centres are to engage themselves in experimental research work and to take part in the framing and adoption of rules to enable the control of quality in production. Each centre is to be controlled by an administrative *Council* whose constitution must be approved by the Minister of Labour.

The income of these centres is to be provided by a compulsory levy from firms within the industry concerned, and from payments made for investigations sponsored by individual firms. The method of collecting these obligatory contributions from the industry is to be arranged by consultation between the *Council* and the Minister of Labour, and the appropriate Ministries responsible for finance.

Provision is also made for the setting up of industrial research centres which may cover a sphere of activity overlapping more than one industry. Such a research centre would be supported, not by contributions from individual firms as in the case of other centres, but by sums contributed by the research centres representing the different industries concerned.

Any research body already in existence and associated with a particular industry may, under the present legislation, make application to assume the status and privileges of an industrial research centre provided that sufficient support is forthcoming from the industry. A research centre which is considered no longer to have the support and confidence of its parent industry may be dissolved with the approval of the Minister (*Bull. British Cast Iron Res. Assn.*, 1948, 9, 251).

### UNESCO Grants

THE *UNESCO* HAS ASSIGNED £60,000 to its work of educational rehabilitation in war-devastated countries. The beneficiaries will be Austria, Czechoslovakia, Greece, Hungary, Italy, Poland and devastated British Colonies.

A grant of \$7,500 has been made to the *International Council for Philosophy & Humanistic Studies* to help the *Council* in the early stages of its work. The *Council* is to co-ordinate the dissemination of information regarding works published or begun in the field of philosophy, human-



istic studies and related branches of study and to encourage the setting up of international organizations in fields in which no such organizations exist.

### F.A.O. Liaison Committee

TO ASSIST THE GOVERNMENT OF India in the discharge of the obligation accepted by them as members of the F.A.O. of the United Nations and to co-ordinate the collection of information required by the F.A.O. from India, and, in general, to act as a clearing house for the exchange of information between the F.A.O., the Ministries of the Central Government, Provincial Governments and Indian States, it has been decided to set up a *National Food & Agriculture Organization Liaison Committee*. It will consist of the Agriculture Secretary to the Government of India as *Chairman* and 16 other members. Of these 5 non-officials will represent the Central Legislature, 2 will be representatives of the *Federation of Indian Chambers of Commerce & Industry*, 3 of the rural peoples' interests to be nominated by the Agriculture Minister, and one representative each from the Ministries of Agriculture, Food, Health, Commerce, External Affairs and Commonwealth Relations. The Vice-Chairman of the *Indian Council of Agricultural Research* will also be a member of the Committee.

### Indian Institute of Chemical Engineers

THE *Indian Institute of Chemical Engineers* (President: Dr. Hiralal Roy) was inaugurated in December 1947 at Patna. At the first session of the *Institute*, also held at Patna in January 48, 11 technical papers were read by members and discussed. Since then, a number of meetings have been organized at Calcutta for the discussion of papers on chemical engineering topics. The *Institute* has over 100 members on its rolls, and its officers are temporarily located at 5/2, Garlick Place, Calcutta.

The first general meeting and the second annual session of the

*Institute* will be held at Allahabad in January 1948 during the *Indian Science Congress Week*.

### ANNOUNCEMENTS

DR. M. S. KRISHNAN, Superintending Geologist, *Geological Survey of India, Southern Circle*, has been appointed Director, *Bureau of Mines*, Government of India, in place of Dr. D. N. Wadia who has retired from December 1, 1948.

THE *Council of the Institute of Engineers (India)* have nominated Dr. D. R. Malhotra as a member of the recently constituted Advisory Council for the *School of Mines & Geology*, Dhanbad, for a period of 3 years.

The Government of India have decided to raise the standard of the *School of Mines & Geology*, Dhanbad, to that of the *Royal School of Mines*, London.

THE UNITED STATES MEDAL FOR Merit, the highest award made by the President of the United States of America, has been presented this year to Sir Alexander Fleming and to Sir Howard Florey for their work on penicillin.

*Universities Commission*: THE Education Ministry of the Government of India have appointed a *Universities Commission* to study the existing system of higher education in India and to submit a plan for its expansion and improvement. The *Commission* will consist of 9 members with Dr. S. Radhakrishnan as Chairman. Two of the members are from U.S.A.—Mr. Arthur E. Morgan, lately President of the Antioch College and Chairman of the T.I.A., and Dr. John J. Tigert, lately Commissioner of Education in the U.S.A. and President of the Florida University. A third member, Dr. Duff, is from Britain. The Indian members, besides the Chairman, are Dr. Zakir Hussain, Dr. Meghnad Saha, Dr. Birbal Sahani, Dr. A. Lakshmanaswami Mudaliar and Dr. Tara Chand.

GERMAN SCIENTIFIC BOOKS: Books published in Germany may now be ordered through a book-seller who may apply directly to the publisher in Germany. The transaction is administered through the *Joint Export & Import Agency of Military Government* and payment can be made in the currency of the country in which the original order is placed. Many publishers in Germany who force a demand outside for a book in Germany generally obtain the required licence soon after the appearance of the book in Germany.

The publication of a new book, *Neure Probleme der Abstammungslehre* by Dr. Bernhard Rensch (Published by Ferdinand Enke Verlag, Stuttgart) has been announced (*Nature*, 1948, 162, 562).

SCIENTIFIC JOURNALS AVAILABLE in India: Dr. Alexander Wolsky, Principal Scientific Officer, Science Co-operation Office for South Asia, University Buildings, Delhi, writes as follows: "It has been suggested that a list of scientific periodicals available in different institutions in India would be very useful for the scientific workers. On account of lack of information as to the availability elsewhere within the country of particular scientific literature, progress in work is often hampered. It has been proposed that the work of compilation of a list of periodicals and journals, which one may consult at a short notice from the libraries in India, may be taken up by this Office. This information will also be very welcome and useful for workers outside India in the region of this office, like Burma, Malaya, Siam, as also the regions of Middle East and East Asia where UNESCO is keeping similar offices."

Dr. Wolsky will be glad to have lists of journals and periodicals available in the libraries of various institutions, grouping them under the following heads: (i) for consultation on the spot, i.e. not to be taken outside, and (ii) for loan (on pre-payment of postage for sending the journals).



# Reports from States & Provinces

## MADRAS

### Technical Training Establishments

THE AUTO SCHOOL FOR TRAINING technicians in automobile technology, the first of its kind in the Province, was recently declared open by the Hon'ble Minister for Industries in Madras. The school will impart training in all branches of automobile engineering and technology. The school is situated in General Patters Road, near Mount Road, Madras.

There are 8 polytechnic centres working in different parts of the Province. A trades school is attached to the polytechnic at Mangalore, and industrial schools are attached to other polytechnics except those at Coimbatore and Vuyyuru. Subjects such as cabinet-making, carpentry, general mechanics, book-binding and blacksmithy are taught in these schools. The courses vary from 2 to 3 years at the end of which certificates are awarded to successful candidates.

A scheme for special training for textile workers is being examined by the Director of Industries.

### Irrigation Project for Malabar

THE FIRST MAJOR IRRIGATION project for Malabar, the Malam-puzha Scheme, has been sanctioned by the Government. The main object of the project is to stabilize the existing irrigation facilities afforded to 40,000 acres in Palghat taluk and its surrounding areas. At a later stage, this scheme will be developed into a hydroelectric project. The scheme will cost Rs. 3.8 crores in its first stage, of which Rs. 135 lakhs will be paid by the Government of India.

The scheme will enable 5,800 additional acres to be brought under cultivation; 7,100 acres of single-crop land can be converted into double-crop land, and on 11,350 acres of double-crop land, a third crop can also be cultivated. As a result of this project, 15,000 tons of rice, in

addition to what is already grown in the area, will be available.

Another project, the Cherumangalam Irrigation Project, estimated to cost about Rs. 8 lakhs and irrigate 1,800 acres, has been sanctioned by the Government.

### Phosphate Deposits

A SURVEY OF THE MINERAL deposits of the Trichinopoly district, carried out in 1940 by the *Geological Survey of India*, reported the occurrence of phosphatic nodules over an area of 11 to 12 square miles. The reserves, occurring to a depth of 50', were estimated at 2,000,000 tons.

The Government have examined the question of utilizing these phosphatic nodules for the production of superphosphates. 3 methods have been suggested for trial: (1) conversion of the ground nodules into superphosphate by treatment with sulphuric acid; (2) calcination of the ground nodules with felspar, quartz and magnesite in a rotary kiln to yield a sintered product in which part of the phosphate in the original mineral is rendered soluble; (3) fusion of nodules with silica and magnesia in electric furnace to yield a product in which all the phosphate is converted into a soluble form.

## NEW DELHI

### Asian Regional Commission of the I.M.O.

THE 11-DAY SESSION OF THE first *Asian Regional Commission of the International Meteorological Organization* concluded in New Delhi on 27th November, 1948, after passing 44 resolutions and recommendations for the better co-ordination and utilization of meteorological services in the region.

The Conference resolved that sub-continental broadcasting centres should be established at New Delhi, Tokyo and Kahbrok (USSR). These broadcasts are intended for the meteorologists of the respective countries and

will be in codes approved by the I.M.O. It was recommended that all countries in the region should organize their own national broadcasts on proper standards so that the sub-continental transmissions are facilitated. The Conference also recommended that national broadcasting centres be established in Afghanistan, Arabia, Iran, Saudi Arabia and Syria. Some of the other recommendations made by the Conference relate to the co-ordination of the national and sub-continental broadcasts, data to be included, codes to be used and standards of transmission. Area for meteorological reports from ships and for shipping forecasts were also allotted.

It was resolved to establish Standing Sub-Commissions for agricultural meteorology, hydrology, meteorological transmission and development of mechanical statistical methods. These sub-commissions will function in close collaboration with the I.M.O. Commissions and co-ordinate and develop the work of the various organizations in the region.

Other resolutions and recommendations passed by the Conference were in respect of improvement of the existing network of meteorological stations and their working. It was decided that the countries in the regions should adopt 4 international synoptic hours of observation for surface observations. It was recommended that 03:00 and 15:00 (G.M.T.) should be adopted for radio-sonde observations (with a tolerance of  $\pm$  one hour), and 15:00 G.M.T. and 00:00, 09:00, 15:00 and 21:00 hours for pilot balloon observations. The adoption of these hours is to come into force from January 1, 1949 and, if that is impossible, by January 1, 1950.

The Conference also recommended that countries in the region should individually or jointly arrange for the establishment of island weather stations, stationary weather ships and meteorological reconnaissance flights and for the securing of weather reports regularly from transport air-craft.

It was found that the international system of storm-warning signals was not adequate for tropical regions and the Conference accordingly recommended a review of the existing system. It was also decided that four countries should be selected for the maintenance of standard barometers to facilitate intercomparisons.

# INDIAN PATENTS

The following is a list of a few of the Patent Applications notified as accepted in the *Gazette of India*, Part II, Section I, for November 1948. Patents from the *Council of Scientific & Industrial Research* are indicated by an asterisk.

## Chemical & Allied Products

### Paints, Varnishes & Lacquers

- \*38062. SIDDIQUI: Conversion of Bhilawan Shell liquid to a non-vesicating drying product for the manufacture of air-drying varnish compositions: *Copolymerizing Bhilawan Shell liquid with turpentine.*
- \*38064. SIDDIQUI & DHAR: Conversion of Bhilawan Shell liquid to a non-vesicating drying product for the manufacture of lacquer varnish, stoving enamels, waterproofing or like materials: *Copolymerizing vegetable oils, Bhilawan Shell liquid, turpentine or rosin.*

### Inorganic Chemicals

- 39320. SUTANE, SALETRE & MENE: Activation of bauxite for bleaching vegetable oils and fats: *Finely powdered bauxite is dehydrated at a temperature of above 300°C., cooled, treated with acid, washed and dried.*

### Organic Chemicals

- 39593. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ: Extractive crystallization process: *Contacting mixture of organic compounds with urea in presence of water, separating the slurry of complex compound formed and decomposing it.*
- 38939. CIBA LTD.: Manufacture of new amines. (Addition to No. 36267): *Reaction of 1:2-dihydrofluoranthene with an alkyl halide containing a basic group in the presence of an agent capable of eliminating hydrogen halide.*
- 39476. MEAD JOHNSON & CO.: Process of preparing 2-sulfanilamidopyrazine: *Reacting 2-aminopyrazine with p-acetaminobenzene-sulfonyl chloride and removing acetyl group from the product.*
- 37345. WILLIAMSON: Method for producing 5, 6-dimethoxy-8-amino quinoline: *Comprises of reacting 4-acetamino-5-nitro-veratrole, glycerine, arsenic acid and sulphuric acid.*
- 40090. I.C.I. LTD.: New quinoline derivatives: *Reacting pyrimidulamino quinoline with a quaternary salt-forming agent.*

### Miscellaneous Chemicals

- 38823. RAO & SREENIVASAYA: Production of distillery washes having alcohol concentration of 10 to 14 per cent by volume: *Fermenting sugar solution by yeast of the species of *Saccharomyces cerevisiae*.*
- 36961. UNIVERSAL OIL PRODUCTS CO.: Process for producing hydrogen: *Reacting catalytically gaseous hydrocarbon and steam.*
- 40111. KLOPPENBURG: Process for winning sugars from the molasses from a cane and/or beet sugar factory: *Alcohol-water solution of molasses treated with lime to form monocalcium salt of the*

*sugar which is thereafter treated with carbon acid to get free sugar and calcium carbonate precipitate.*

- 31398. I.C.I. LTD.: Construction of aircraft runways, roads, floors, and like surfaces and manufacture of water-repellent materials therefor: *To the earth's surface is applied an aqueous solution of ammonium or other water-soluble salt of a water-insoluble carboxylic acid and an aqueous solution of a water-soluble multivalent metal salt.*
  - 31610. I.C.I. LTD.: Manufacture and application of water-repellent constructional materials: *Preparations are intimate mixtures in powder form containing at least two ingredients from the following, viz. (a) water-soluble salts of water-insoluble carboxylic acids; (b) water-soluble salts of polyvalent metals; (c) water-insoluble polyvalent metal salts of carboxylic acids obtainable by interaction of (a) and (b); (d) water-insoluble carboxylic acid; (e) salts of acidic polyvalent metal oxides, which salts are either water-soluble or decompose on contact with water into a water-soluble base and a water-insoluble polyvalent metal hydroxide, subject to certain provisions.*
  - 36682. KHORANA & BHUNVARA: Mucilage of plantago as an emulsifying or thickening agent. *Emulsifying paraffin or vegetable oils with extract of plantago seeds.*
  - 37604. ANGLO-IRANIAN OIL CO. LTD.: Synthesis of hydrocarbons and oxygen-containing compounds from mixtures of carbon monoxide and hydrocarbon. *Reacting carbon monoxide hydrogen and olefine in presence of catalyst.*
  - 39388. N. V. VEENENDAA'SCHE SAJET — EN VIJFSCHACHTFABRIEK VOORHEEN WED. D. S. VAN SCHUPPEN & ZOON: Process for the production of sterols and of their additions products from material containing sterols. *Treating sterol containing material with compounds containing NH<sub>2</sub>-groups and decomposing the addition product.*
- ## Fuels & Lubricants
- 38266. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ: Mineral greases containing barium and/or strontium soaps: *Comprises of using oxy-fatty-acid in mineral oil.*
  - 37719. STANDARD OIL DEVELOP. CO.: Process for the gasification of carbonaceous materials and for the manufacture of water gas: *Withdrawing a portion of carbonaceous material from the reaction zone and contacting it with air under combustion condition.*
- ## Medical Research & Practice (INCLUDING CLINICAL APPLICATION OF DRUGS & PHARMACEUTICALS)
- 38479. GHOSH: Process for the preparation of cinchonine ethyl carbonate and dichinonine carbonate: *Condensing cinchonine with car-*

*bonyl chloride or ethyl chlorocarbonate or diethyl carbonate.*

37354. CAMPBELL : Antimalarial compounds, intermediates and process of producing them : *Condensing O-nitro-p-alkoxy aniline with methyl-vinyl ketone to form 6-alkoxy-8-nitrolepidine and condensing the lepidine after reduction with alkylaminoalkyl halides or their hydrohalide.*

39684. DIRECTOR, HAFKINE INSTITUTE : A process for the preparation of 2-(para-aminobenzene-sulphonamido)-thiazole : *Condensing p-acetamino-benzene-sulphonyl chloride with 2-amino-thiazole in the presence of alkali carbonate and heating the condensation product with a further amount of 2-amino-thiazole.*

#### **Metals & Metal Products**

37018. JOHN MILES & PARTNERS (LONDON) LTD. : Improvements in and relating to the refining of metals : *By projecting oxidizing fluids into the molten metal.*

37606. UNITED ANODISING LTD. : Production of anodic films on metal surfaces : *Current passed through metal articles disposed between anode and cathode but not connected to them.*

38302. UNITED ANODISING LTD. : Electrolytic treatment of metals : *Bath containing orthophosphoric acid, organic inhibitor, water and sulphuric acid, used for polishing metal put as anode.*

39720. REPUBLIC STEEL CORPN. : Refining of ore in blast furnaces : *Ratio of charge maintained between 2.45 and 2.8, blast rate kept above normal, and discharge gases throttled to maintain furnace top pressure at least 5 lb. per square inch gauge.*

#### **Miscellaneous**

38633. CHLORIDE ELECTRICAL STORAGE CO. LTD. : Level indicating devices for electric accumulators : *Of the type comprising a float with a stem projecting upwards with marking thereon.*

37448. BOOTH : Aeration or flotation machines and methods of froth flotation : *Setting in motion an axial current of aqueous pulp, intercepting the pulp flow along an imperforate plane having a trailing edge and sweeping a current of aerating fluid over and aerors the trailing edge into the pulp flow.*

38424. BAKE : A method and an apparatus for withering tea-leaf : *Tea-leaf is conveyed by means of an air current through one or more withering rooms, and subjected to a movement opposite to the direction of said current.*

39767. APTE & SENGUPTA : An improved type of gas and air filter : *An air filter for internal combustion engines in which saw dust is used instead of coir, felt, etc.*

38046. FREYSSINET : A method of manufacturing pre-stressed concrete articles particularly railway sleepers and articles : *Placing in the rounded end portions of hoops two members capable of resisting the tension exerted by these hoops, incorporating the said members in the interior portions of a mould and housing concrete into the mould.*

39930. M. KONECNY : Manufacture and application of insulating material for buildings and the like : *Consisting of bands made by binding corncobs.*

## Study of the History of Science

THE decision to form a Society for the promotion of study and research in History of Science in India, reached at a meeting of Indian scientists at Allahabad on January 3, is to be welcomed. Such an organization co-operating with the International Union of History of Science would give a much-needed fillip to the promotion of knowledge on a subject of the utmost interest and importance to this country.

It must be admitted that the attention given so far to the study of history of sciences in India is very meagre, but then the recognition of history of science as an independent discipline and the realization of its importance as a subject for study and research are comparatively recent. It is true that both history and science are ancient, but their combination, "the application of the well-known methods of history to the well-known facts of science" is relatively new. It is only since the close of World War I that the subject gained a place in the curriculum of studies in American universities, and at a somewhat later date in British universities. The first journal devoted to history of science—the *Isis*, was started in Belgium in 1913, and the History of Science Society, an international organization with its centre in America, was founded later. In India, while individual scientists have made valuable contributions to this subject, its study at the university level was initiated only 5 years ago in the Delhi University, thanks to the foresight of the members of the Faculty of Science who were seized of the importance of its study, particularly in India, which shares with Egypt and early Asian civilizations the distinction of being the ancient home of science.

It is of interest to examine briefly the value of this comparatively new discipline in order to appreciate the significance of the decision taken at Allahabad to establish the new Society. History of science, as we

have stated before, is both history and science. Science is systematized positive knowledge—or what has been taken as such in different ages and in different places. In the words of Dr. George Sarton\*, the accumulation and systematization of positive knowledge are the only human activities which are truly cumulative and progressive, and history of science is the only history which can illustrate the progress of mankind. In fact, according to Sarton, *progress has no meaning in other fields than in the field of science*. The understanding of this progress is greatly assisted if we penetrate into its genesis and evolution. "Knowledge is not something dead and static, but something fluid, alive and moving. The latest results are like the new fruits of a tree; the fruits serve our immediate practical purposes, but for all that it is the tree that matters. The scientist of philosophic mind is not interested so much in the latest results of science as he is in its eternal tendencies, in the living and exuberant and immortal tree. The fruits of today may be tempting enough, but they are not more precious, to his way of thinking, than those of yesterday or tomorrow." Early science was once alive, even as our own will soon be dead, and to understand the life of science we must observe it as it grows around us.

And why should there be so much emphasis on the *understanding* of science? Science represents the greatest triumph of the human mind. Its positive achievements are many, and it has provided the means to advance our material welfare. Even more important than its achievements are its methods and techniques. It is of fundamental importance that every man and woman should understand the methods and spirit of science and should be educated to consider everyday problems and social and

\**The Study of the History of Science*, 1936, Harvard University Press, Cambridge, Massachusetts.

political controversies objectively and disinterestedly. It is this objective, disinterested struggle for the discovery of truth that forms the theme of history of science. The great men and women to whom we owe our heritage of knowledge and culture are the heroes of this history. Their biographies, which are of intense human interest and provide material for the study of man, fill its pages. They are the actors in the great and noble struggle for knowledge, and it is they that fashioned the tools which mankind employed in raising itself from one cultural level to another. The method of history is the method which leads to the understanding of science — the method which leads people to appreciate the tasks and social implications of science.

The promotion of the study of history of science is in effect the promotion of a wider understanding of science and scientific method, and it is for this reason that we welcome the inauguration of the Society. Many schemes have been formulated and implemented for the development of scientific research in this country. These are of the utmost value for the promotion of national welfare. But the

problem of educating the people at large to appreciate the objects and methods of science so that they may enthusiastically support scientific research and development has yet to be tackled. History is an instrument of public education, and we look upon the formation of the new Society as a landmark in the progress of science in the country. Professor Govind Behari Lal, Science Editor of the Hearst group of papers in the United States, during his recent visit to India, urged the need for a scientific movement in this country. "We have got to infuse the masses with the scientific spirit;" he said, "they should have a scientifically trained mind. In the past our conditions taught us to think in terms of politics. We have now to learn to think in terms of science." The scientific movement to which Professor Lal has referred has been, in our opinion, well and truly initiated by the formation of the Society of History of Science, and we have every confidence that the new Society would help in a wider understanding of the tasks of science and lead the people of this ancient land with its rich scientific traditions "to think in terms of science".

## Central Food Technological Research Institute

THE Cheluvamba Mansion, a beautiful and spacious building situated on 160 acres of land in the capital city of Mysore, has been placed at the disposal of the Government of India for the location of the Central Food Technological Research Institute. The mansion was formally taken over by Pandit Jawaharlal Nehru on behalf of the Government of India on December 29 at a special function organized for this purpose and attended by a large gathering of scientists and prominent State officials.

The Cheluvamba Mansion is situated on elevated ground in close proximity to the Mysore Railway Station and commands an impressive view of the Chamundi Hill on one side and the main part of the city on the other. The mansion is a 2-storeyed building measuring 210' by 445'. There are over 100 halls and rooms covering a floor area of about 130,000 sq. ft. The halls are ideally suited for lecture halls, library, machine rooms and

workshops, while the rooms are spacious, well ventilated and can be readily converted into laboratories. The gardens and roads are well laid out. The surrounding ground is suitable for construction of additional laboratories and staff quarters. It is proposed to make immediate arrangements for converting a part of the mansion into laboratories and to start work in the summer of 1949.

The Food Technological Research Institute is one in the chain of eleven national institutions sponsored by the *Council of Scientific & Industrial Research*. The Institute will have divisions for research and investigation on storage and reclamation of food grains, composite and emergency foods, food engineering and dietetics, adulteration, etc. Steps would be taken to stimulate food industries in the country, and the Institute would collect food statistics and issue bulletins on food and food products for the information of the public.

# Characteristics of the Ionosphere at Calcutta ( November 1948 )

S. S. BARAL, S. N. GHOSH, R. K. MITRA,  
D. C. CHOUDHURY & ( Miss ) T. K. PAL

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THE following are the ionospheric data for the month of November 1948. The observations were made at each hour of the day for 5 days a week.

Fig. 1 presents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form during the month of November 1948. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during February 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when the E region was found to be abnormal and the corresponding penetration frequencies and heights. It is found that in this month the abnormal E ionization is most prominent during the early morning hours.

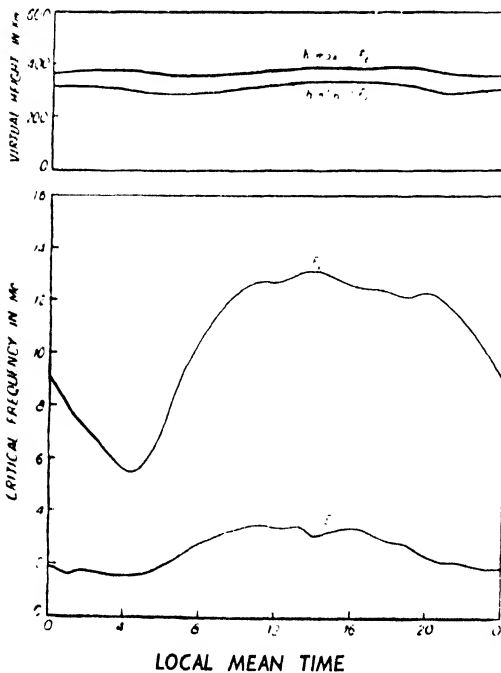
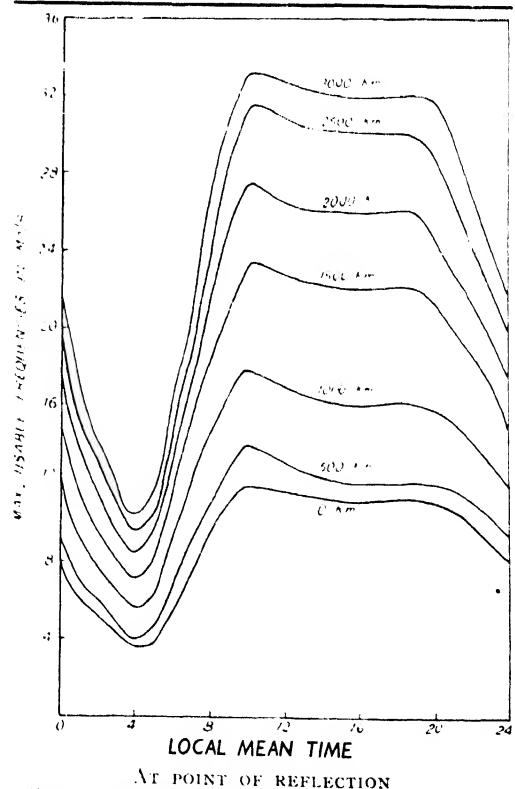


FIG. 1 — NOVEMBER 1948. 5 HR. 54 MIN. AHEAD OF G.M.T.

MONTH & YEAR	DATE	HOURS	$f_oF_2$ Mc	$h'_{F_2}$ Km.
Nov. 1948	5	21.00	3.00	90
		22.00	2.00	90
		23.00	2.00	90
	6	00.00	2.00	90
		01.00	1.96	90
		02.00	2.00	90
		03.00	2.00	90
	9	01.00	1.80	90
		02.00	1.85	90
	23	02.00	2.40	90
		03.00	2.35	90
		04.00	2.10	90
	26	02.00	1.80	90
		03.00	2.00	90
		04.00	2.50	90
	27	22.00	2.50	105
		23.00	2.00	105
	28	02.00	2.50	90
		01.00	3.10	105
		02.00	3.35	105
		03.00	3.50	90
		04.00	3.30	90
		05.00	3.00	90



AT POINT OF REFLECTION  
FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION VIA  $F_2$  LAYER, FEBRUARY 1949.

# Characteristics of Photoflash Lamps

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**P**HOTOFLASH lamps first appeared in the market in 1930. The bulb was filled with oxygen and contained a quantity of aluminium foil which was flashed by igniting a chemical covering a thin wire through which a current was passed<sup>1</sup>. As the total duration of the flash was only a small fraction of a second, it was immediately accepted as a suitable source of illumination for indoor, instantaneous photography. The bulb was naturally a great improvement over the magnesium powder whose open firing often caused accidents, apart from the uncertainty of the flash itself<sup>2,3</sup>. The photoflash lamp, on the other hand, provided a completely controlled source of illumination.

To begin with, a certain amount of judgement was necessary in taking a photograph. The shutter was opened just before the flash and closed immediately afterwards. This naturally entailed some practice before successful photographs could be taken, and to get over this difficulty "synchronizers" were introduced. These were small mechanisms which permitted simultaneous and automatic opening and closing of the shutter at the time of the peak intensity of the flash. This maximum intensity was standardized to occur after about 20 milliseconds of the switching on of the current in the filament. Under such conditions the total duration of the flash after reaching half peak intensity was slightly less than 10 milliseconds. As it was found difficult to synchronize the opening of the shutter with the flash during such a short interval, attempts were made to increase the duration of maximum brilliancy of the lamp. This was achieved by replacing the foil with shredded foil or filaments. The duration of the peak intensity was, in this way, doubled and led to an easier manipulation of the shutter<sup>4</sup>. Curves in Fig. 1 show the chief characteristics of the foil-filled and filament-filled lamps. It will be seen that both types reach the maximum peak intensity about the same time. But whereas the foil-filled lamp reaches a higher peak intensity than the filament-filled lamp, the

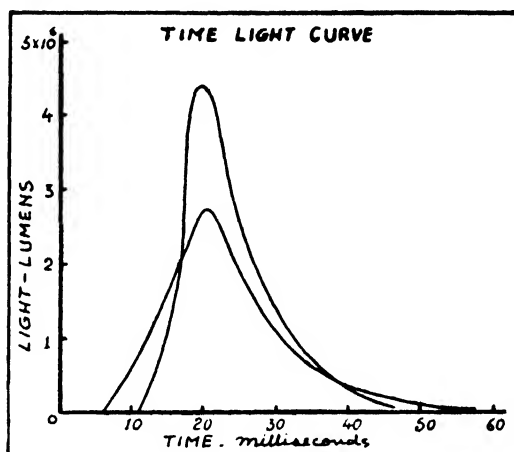


FIG. 1

duration of the flash is larger in the latter. Also the flash starts more quickly in a wire-filled lamp.

The suitability of the radiant energy emitted by the photoflash lamp for photographic purposes<sup>5</sup> can be ascertained by a comparison of colour-temperatures (temperature for black body giving the same colour) of various sources (TABLE I). The photoflash lamp is the nearest approach to sunlight.

An interesting recent development of the flash bulb is the *GEC* (speed, midget) flash bulb. This does not contain any wire or foil-filling. The sole illuminant is an extra large dose of primer compound on the heating wire. The special advantage of this is the elimination of time lag.

The foil-filled lamps investigated by us were made in the laboratory, for the first time in India, with materials available here.

TABLE I

SOURCE	COLOUR TEMPERATURE °C.
Nernst filament	2,400
Tungsten temperature	2,780-3,000
Flash powder	3,800
Carbon arc	4,000
Photoflash lamp	5,000
Sun	5,400

Our studies have shown that the bulbs compare quite favourably with the imported ones.

### Construction

The essential constituents of the lamp are: (a) hollow bulb of known capacity; (b) hair-thin filament connected to electrical leads; (c) primer compound coating for the filament; (d) aluminium foil-fillings; and (e) oxygen at suitable pressure.

The bulbs were supplied by *Kaycee & Co., Ltd.*, Lahore, and were those ordinarily used for lighting purposes. To avoid accidents, particularly while dealing with high-pressure oxygen flash lamps, it is desirable to give them an inner coating of transparent lacquer. This, however, was not necessary in the present work since the pressure of oxygen was always kept much below than that of the atmosphere. The glass should be of uniform texture to minimize chances of cracking.

As regards the filament for coating the primer compound, nichrome was selected as the most suitable. It has high electrical resistance combined with a high melting point ( $1400^{\circ}\text{C}.$ ). Since only a few millimeters of the wire are employed, the gauge number should be so selected that even one or two volts from a cell could provide sufficient current to raise the temperature of the wire to a degree sufficient to cause the ignition of the primer compound. SWG No. 40 (resistance 94 ohms/meter) was used. Half a centimeter of this wire has a resistance slightly less than half an ohm. Since the fusing current is 1 amp., one to two volts should be enough to cause fusion.

**Primer Compound**—A mixture of sulphur, charcoal and nitre was used in the proportion 2:1:1. This was coated on the filament with a paste of shellac in methylated spirit. The mixture burns with a fountain effect, producing sparks and aids in the complete combustion of the foil contents of the bulb.

**Aluminium Foil**—The thickness of the foil in the imported bulbs is of the order of 0.00004 cm. The foils used in the present work varied in thickness from 0.000056 to 0.00012 cm. as measured by weighing foils of known areas. These foils were locally produced by hammering in gold-beaters skin and were found quite satisfactory.

**Oxygen**—The actual quantity of oxygen required depends upon the weight of the aluminium foil used. For complete combustion the bulbs are filled with dry oxygen at a pressure of about 30 cm. of mercury for 65 mg. of aluminium charge. Oxygen

was obtained from commercial suppliers and dried by storing over calcium chloride. The jar containing calcium chloride is first evacuated and then filled with oxygen at atmospheric pressure. It gets sufficiently dry after 24 hours. The reservoir is connected to the bulb and sufficient oxygen admitted to give the desired pressure as indicated by a mercury manometer.

### Characteristics of the Lamp

The following characteristics of the bulb must be known: (a) total quantity of light in lumen-seconds; (b) maximum intensity of the radiation in lumens; (c) time from the application of the filament current to the start of the flash, in milliseconds; (d) Time from the application of the filament current to the peak intensity of the flash, in milliseconds; (e) total duration of the flash above a certain specified minimum intensity; and (f) spectral distribution of the flash light.

The bulbs are manufactured in different sizes. A determination of the characteristics of the bulb fixes the exact use to which a bulb of any particular size can be put to the best advantage<sup>5</sup>. For example, the total quantity of light of commercial bulbs manufactured by the *General Electric Company* and numbered 10, 20 and 75, are suitable for portrait photography, small groups and large groups respectively. (c), (d) and (e) characteristics are used to regulate the speed of the camera shutter in order to avail of the maximum light in the flash. Since the opening of the shutter is not instantaneous, the actual quantity of light across will vary with time and may be represented by a graph of the type shown in Fig. 2. The shutter opens fully only for a part of the time and, consequently, the shutter and the flash should be so synchronized that the flash reaches the peak at the point A, when the shutter is fully open.

The determination of the spectral distribution of the flash is useful for colour photography<sup>6</sup>. Slight variations in the distribution do not matter for ordinary photography, but as the distribution plays an important rôle in the colour photography, due consideration should be given to it.

### Rotary Flashometer

For determining the characteristics mentioned above a flashometer is employed. Various forms of this have been used by previous workers<sup>7-9</sup>. The rotating flashometer



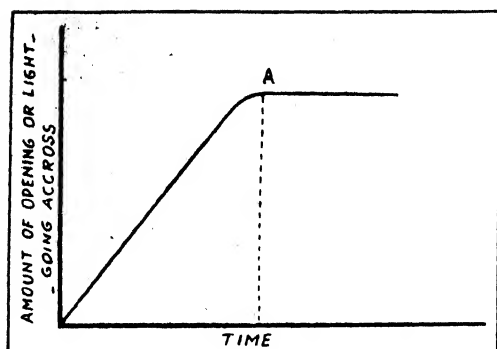


FIG. 2

used in the present experiments was designed and constructed in the laboratory and has proved completely satisfactory. This consists of a rotating drum on which a photographic film is wrapped. The light from the flash bulb, after passing through a slit, is made to fall on the film and traces out a band of graded intensity. Starting with low intensity it reaches a maximum and then again falls to zero. As the drum rotates at a previously determined speed, the total duration and time for which the lamp flashes at its peak value can be ascertained. Absolute intensity can be measured by comparing with a standard source. A special feature of the arrangement was the commutator by means of which it was possible to arrange the opening of the slit just before the closing of the electric circuit for the flash, and the closing of the slit automatically when the bulb ceased to glow. Details of the assembly are as follows.

**Camera** — This consists of two parts: a wooden box of special construction and a metallic drum capable of rotating round its axis, held inside the box. The box is  $14'' \times 6'' \times 6''$  with about a dozen circular holes, each  $0.25''$  diameter, in a line on one of its sides. Attached to this side of the box is a metallic cover which is worked by an electromagnet for opening and closing the holes, and which can be put in the front of any one of the openings. The drum is  $4''$  in diameter and  $12''$  long, and is positioned at the centre of the box by an iron rod running along the axis of the drum. A part of the rod projects outside the box and supports a pulley for a belt drive for rotation. The surface of the drum on which the photographic film is wrapped is about an inch away from the holes in front of the box. In order to get a sharp impression on the photographic record a small metallic cylinder with a sharp slit

attached to the side is inserted in each one of the small openings on the side of the box, and the slit brought as close to the drum surface as possible.

The electromagnetic arrangement for opening and closing the holes is shown in Fig. 3. E is the electromagnet which actuates the rod MN about the fulcrum L. H is the hole in the metallic strip. The hole normally remains closed, and opens only when the magnet is actuated. By a spring action the slit closes again as soon as the current is stopped.

**Commutator** — To operate the flashometer it is necessary to have some mechanical contrivance by which the following operations could be automatically carried out successively: Opening of the shutter at pre-determined intervals, flashing the lamp and finally closing of the shutter. This was arranged by a commutator of special design shown in Fig. 4.

This consists of a cylinder  $3''$  in diameter and  $4''$  in length, partly brass and partly ebonite as indicated. A V-shaped spiral groove of 5 or 6 turns is cut over the whole length, the last turn being a continuous circular groove. The commutator is suitably supported and made to rotate by means of

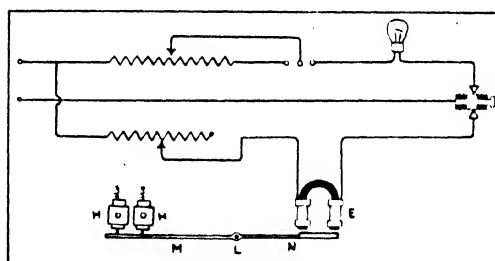


FIG. 3

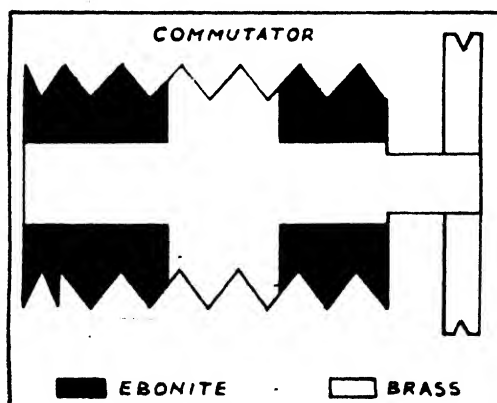


FIG. 4

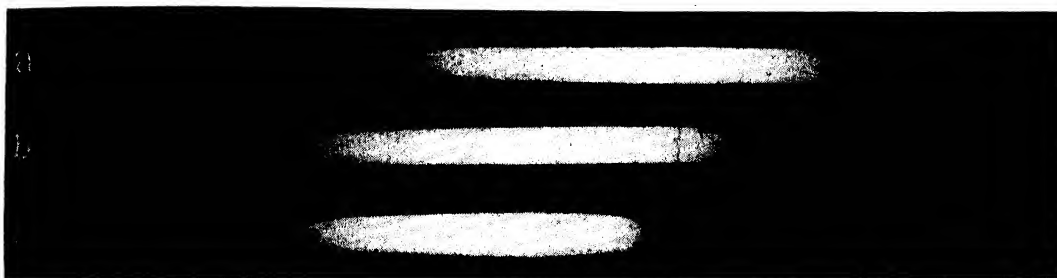


FIG. 5 PHOTOFLASH TRACES : (a) OXYGEN, PRESSURE 25 CM. ; (b) OXYGEN, PRESSURE 30 ALUMINIUM, 60 MG. (c) OXYGEN, PRESSURE 35 CM., ALUMINIUM, 60 MG.

a belt drive at a regulated speed. Two elastic metallic brushes press against the grooves and move laterally as the commutator is rotated. This lateral movement stops as soon as they reach the circular groove. As the brushes move in the spiral groove, one of the brushes leading the other by a fraction of a revolution, electrical connections are made by the metallic portions of the groove. The leading one opens the shutter by actuating a current in the electromagnet, and the other closes the circuit for flashing the lamp. The circuit is broken after one and a half revolutions as soon as the brushes come over the ebonite portion. The circuits are shown in Fig. 3.

Some of the photographic records made with different weights of aluminium foil at various pressures of oxygen are shown in Fig. 5. The total duration of the flash can be estimated from those with a fair degree of accuracy if the speed of the drum is known.

For the determination of the interval of time between the contact and the peak of the flash, a 2-volt torch-bulb is put in series with the flash bulb. The metallic brush used for operating the electromagnet was permanently connected to the metallic part of the commutator through a key. Also two slits are provided instead of one, one for the flash bulb and the other for the 2-volt bulb, both worked by the same electromagnet.

In starting the observations the motor was allowed to run for a time till it acquired a constant speed. The key was then closed and the brush allowed to fall on the commutator. Two flashes are traced out on the film, one through each slit. Assuming that the time required for lighting up the 2-volt bulb is negligibly small, it is easy to determine the interval between the contact

and the peak of the flash. Fig. 6 is a record of one of these observations.

*Measurement of Light Intensity*—For measuring the illuminating power a 500-watt lamp was selected for comparison. Traces on the film with this lamp at different distances were obtained and microphotographed along with that of the flash bulb. For cutting down light from the bulbs by a known fraction, screens for transmitting a definite fraction of the light were used. These screens were prepared by stretching metallic strips of about 2 mm. width at suitable distances across a frame and placing them between the bulb and the slit at the time of taking the trace. No special advantage is obtained by rotating the frame as the duration of the flash is too short.

#### Lumens & Lumen Seconds

The illuminating power of the flash lamp at its peak value is given by the relation

$$4\pi W \frac{D^2}{d^2} \times f \approx \frac{100}{T}$$

where

$W$  = wattage of the standard lamp whose intensity at a distance  $d$  (in feet) equals the intensity of the flash lamp placed at a distance  $D$ .

$T$  = percentage of light transmitted through the screen.

$f$  = conversion factor for expressing wattage in candle powers.

The photomicrogram gives the variation of the intensity of the flash bulb with time. For determining the total output of light in lumen seconds, a time-intensity curve is plotted for the flash bulb. The distances on the trace are convertible into time scale and the deflections of the microphotometer are expressible in terms of intensity; since the response of the microphotometer, as that of the photographing film, is not linearly

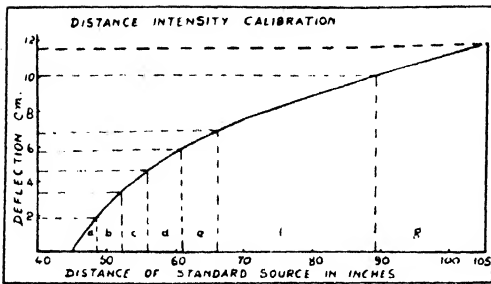


FIG. 6 — DEFLECTION OF THE MICROPHOTOMETER FOR THE STANDARD SOURCE TRACE.

related to the intensity, the curve is divided up into a number of smaller parts where this relation may be considered sensibly linear. The mean percentage of the intensity of light for each one of these parts is then calculated by first obtaining the point by extrapolation where the intensity may be considered 100 per cent and then applying the inverse square law for calculating the average intensity for various deflections. Conversion factors are obtained for time-axis and intensity-axis. The total area in the time-intensity graph being divided into a number of smaller areas, the actual energy output of the flash lamp is given in lumen seconds by multiplying each area with its corresponding conversion factor and adding all the products together ( Fig. 6 ).

The following observations recorded in a typical set are given as an illustration.

TABLE II

DISTANCE IN INCHES	DEFLECTION IN CM.
52	3.5
58	5.2
70	7.5
90	10.5

Illuminating power of the flash bulb at its peak value being

$$Ip = 4\pi W \frac{D^2}{d^2} \times f \times \frac{100}{T}$$

where

$$W = 500 \text{ watts}$$

$$f = 2 \text{ c.p./watt}$$

$$d = 45/12 = 3.7'$$

$$T = 25\%$$

$$Ip = 78 \times 10^3 \text{ lumens}$$

The curve is divided up in seven lengths, as shown in Figs. 6 and 9. The intensity is 100% at 45".

Total length of the flash = 12 cm.

Total duration of the flash = 120 milliseconds.

Most effective duration of the flash = 40 milliseconds.

Lapse of time between contact and peak of flash = 100 milliseconds.

Total energy output =  $172 \times 10^3$  lumen seconds.

Energy output at the peak = 8,500 lumen seconds.

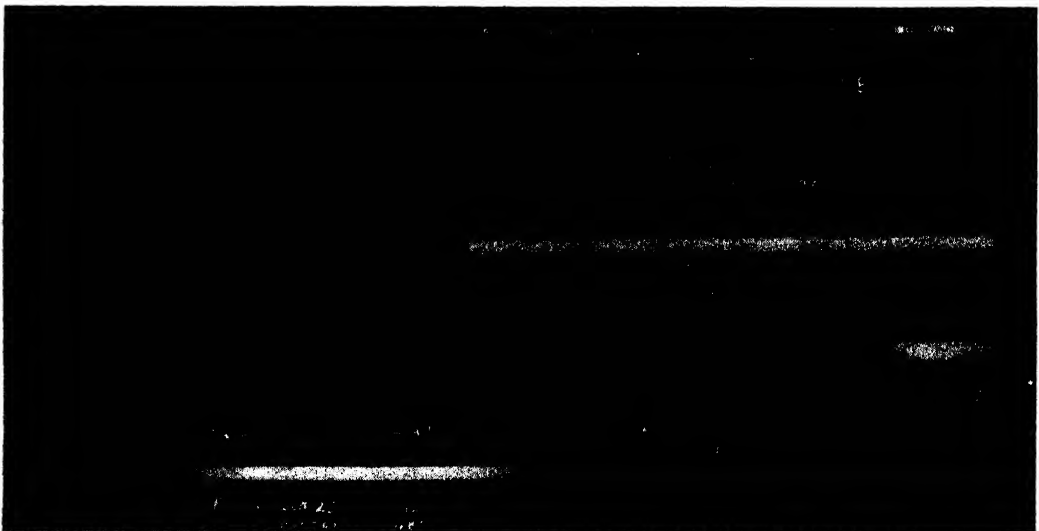


FIG. 7 — TRACE OF THE FLASH BULB.

TABLE III

HORIZONTAL DISTANCE ALONG X-AXIS GIVING TIME CM.	VERTICAL DEFLECTION ALONG INTENSITY CM.
0.0	11.6
1.4	10.1
3.7	4.3
5.2	5.9
7.2	4.0
9.0	3.2
12.5	0.7
12.9	0.6
16.8	0.1
21.8	0.1
25.1	0.3
26.3	0.7
27.0	1.4
28.9	2.5
32.7	5.0
35.7	7.0
38.8	9.9

TABLE IV

No.	DEFLECTION IN CM.	MEAN DISTANCE IN INCHES	% INTENSITY
a	0.0 to 2.0	47.0	91.8
b	2.0 to 3.5	50.5	79.0
c	3.5 to 4.4	53.0	72.0
d	4.5 to 6.0	56.0	60.5
e	6.0 to 7.0	61.5	50.3
f	7.0 to 10.0	78.5	32.9
g	10.0 to 11.5	105	19.8

Spectral Distribution

To examine the character of light in the flash, its spectrum was photographed with a quartz spectrometer. For this purpose a side tube was attached to the bulb and a quartz window provided. This was placed quite close to the slit of the spectrometer. The spectrum of the flash was very diffuse containing arc lines of copper and alu-

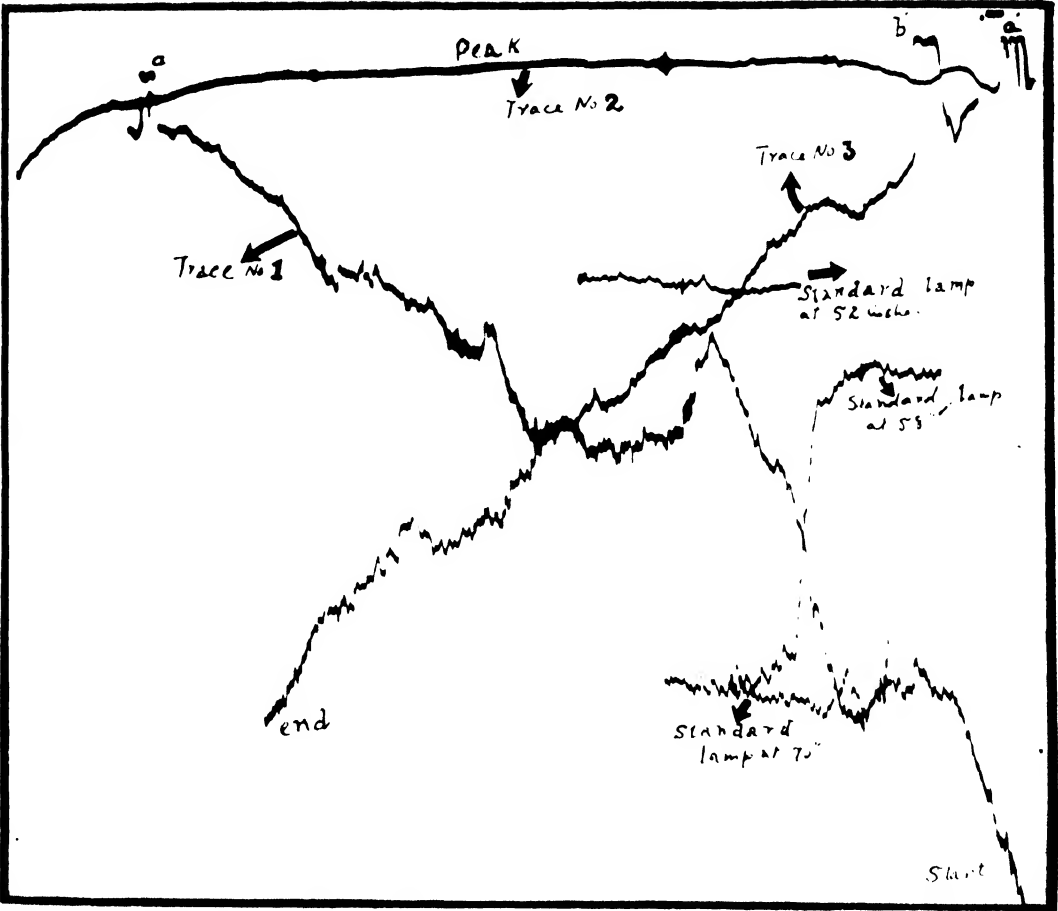


FIG. 8 — MICROPHOTOMETER RECORD ( TAKEN IN THREE PARTS ) DEFLECTION OF THE MICROPHOTOGAM. PHOTOMICROGRAPHS 1, 2 AND 3 ARE FOR THE THREE PARTS OF THE PHOTOFLASH TRACES. REMAINING THREE TRACES ARE FOR THE STANDARD LAMP AT DIFFERENT DISTANCES.

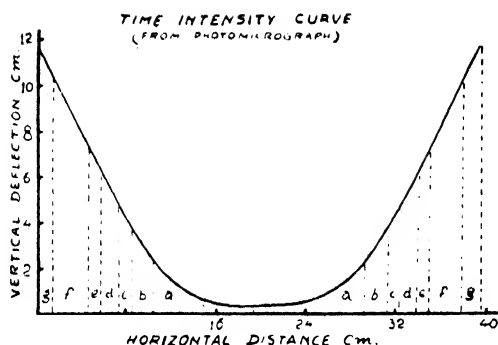


FIG. 9 — DEFLECTION OF THE MICROPHOTOMETER FOR THE FLASH BULB TRACE.

minium superimposed on a continuous background.

In order to see how different wave-lengths in the spectrum change with time, the slit of the spectrometer was reduced to a small square of  $\frac{1}{2}$  mm. side. The telescope of the spectrometer was removed, and the light coming out of the spectrometer prism was focussed by a lens on the drum of the rotating flashometer. The drum was then set rotating and the lamp flashed. The resulting spectrum gave the wave-lengths emitted at different times. It was found that these

were not very different from those observed in the previous photographs.

These observations are, however, only of a preliminary nature and the work is being continued.

The work described in this paper was carried out under the direction and supervision of Dr. P. K. Kichlu, and the writer wishes to express his best thanks to him for his interest in the work and for his encouragement during the investigation.

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# REVIEWS

**Race, Language and Culture**, by Franz Boas (Macmillan & Co. Ltd., London), 1948, pp. xx+647, 90 illustrations. Price 27s. 6d. net.

THE LATE PROF. FRANZ BOAS'S CONTRIBUTION to anthropology is well known both in India and abroad. His work in the fields of physical and cultural anthropology had been prolific, thought provoking and competent. He founded a school, and his students are spread over the Americas and many from Europe and Asia have learnt at his feet. He was one of those who have given status and reorientation to the science of man. To him anthropology had never been merely antiquarian research, mere curiosity, "regarding the early history of mankind". "Knowledge of the life processes", wrote Prof. Boas, "and behaviour of man under conditions of life fundamentally different from our own can help us to obtain a freer view of our own lives and of our life problems." He was more interested in the dynamics of life than in the material of cultural life though the relation between the two was never ignored by him.

In this volume Prof. Boas has thrown light on many problems of anthropology, physical and cultural, and his treatment of the two branches shows his synoptic attitude to culture which finds culmination in the *gestalt* approach to culture by the most articulate of his disciples, Dr. Ruth Benedict. The book is divided into three major sections, Race, Language and Culture, with a small tail of miscellaneous writings on aims and advances in anthropological methodology. The problems mooted and the arguments advanced have the freshness of field and first-hand research; the academic approach, where it has become necessary, is not allowed to be dogmatic, though the egalitarianism in him runs through the whole narrative. It is difficult to swim against the tide and Boas's task has been complicated by the emotional reaction of peoples of his country, and his thesis on the sociology of racism has had to be administered in doses mild and innocuous. Whether his attitude to racism is deliberate or not, his approach has the merit of objectivity which seldom has

inspired the "so-called" literature on race and "racialism". Prof. Boas recognizes the influence of the environment on the body form. "I think", he writes on page 59, "the evidence showing that the form of the head is susceptible to environmental influences is inconvertible." "I also believe that adequate proof has been given for modification in the width of the face under changed conditions of life." Yet Prof. Boas avows that the causes of these changes are still entirely obscure. If chemical changes occur under new environmental conditions and unequally influence growth in different directions, he advocates investigations of the instability of the body under varying environmental conditions as one of the most fundamental subjects to be considered in an anthropometric study of human population. This is the scientific attitude and this is Boasian in the fullest sense of the term.

Boas has been a most difficult personality in ethnology for his achievement, in the words of Prof. Lowie, is full of paradoxes. Boas has been accused of a lack of synthesis though his is a method probably more totalitarian than the functional approach of Malinowski. Says Lowie, "It is still more paradoxical that this indefatigable collector who has consistently preached the totalitarian view of culture, has produced not a single full length monograph of any one tribe." But Boas's training and his interests never compromised with the study of closed groups, while his "totalitarianism" finds expression in the sweep of studies he made his own. His failure to write a monograph is probably due to the recognition of the gaps that every ethnologist has to fill up with cement and plaster, but Boas was too puritanical to attempt the same. Other strong points in Boas's discipline are his historical sense, his relevant arguments on the phenomenon of cultural parallelism which he weighed cautiously with mythological evidences, his analysis of the factors leading to Eskimo migration, his insistence on the comparative method with regard to the display of museum specimens and his substitution of phenomenism for "an arid conceptual realism based on premature

classification", exemplified by the literature on Totemism.

In his approach to cultural evolution, Boas has an enigmatic rôle. While Lowie thinks Boas was not anti-evolutionary but merely attacked Morgan's and others' evolutionary schemes, Leslie White thinks it otherwise. Boas and his students are said to have shown little hospitality to the theory of cultural evolution. It is also true that Boas and his school, even if they did not object to the theory of cultural evolution *in toto*, did not develop an adequate form of evolutionist theory. Goldenweiser and Radin also have characterized Boas's philosophy as anti-evolutionist, a contention which has formed the subject of an animated controversy in the pages of the *American Anthropologist*, more particularly between Lowie and Leslie White. Even if Prof. Boas has not been an anti-evolutionist, his approach to problems of race and culture in effect gives the impression that his first-hand investigations and laboratory experiments did not encourage him to support the evolutionary point of view but the departure probably was the result of a scientific scepticism. Cultural evolution, however, stands on a different footing than that of racial evolution, and as Ellwood has shown, a parabolic development from pre-control to rational decontrol may replace an unilinear evolution which Boas and his school have rightly contested. Whether Boas believed in evolution or not, the march of culture is a process in which evolution does have its rôle and as Leslie White (*Am. Anth.*, 1947, 49, No. 3) affirms, evolution is not losing ground in American anthropology though the Boas school has probably done much to undermine it.

The sweep, the contents, and the methodology that Boas condenses in this volume will benefit students of anthropology in all parts of the world, and although from a cursory perusal the chapters may appear to be "bites", there is a vast store of knowledge at the back of the information doled out. "Boas is a man lacking and scorning any artifice that might attract students. Yet he has trained and influenced the greatest number of professional anthropologists." The book as presented will equip the students of man with multiple starting-points for both field study and academic research without Prof. Boas's claiming the same. We recommend the book to all anthropologists and also to laymen, for

both are likely to profit in obviously diverse ways. The absence of an index is a desideratum for which we can blame the publishers alone.

D. N. MAJUMDAR

**A Human Relations Casebook for Executives and Supervisors**, by Frances S. Drake & Charles A. Drake (McGraw-Hill Book Co. Inc., New York, London), 1947, pp. xiv+187. Price \$2.50.

THIS IS A BOOK WHICH SHOULD BE CAREFULLY read by all persons charged with the responsibilities of running an industry or a business house or any other organization employing a number of persons. In my opinion our industrialists have been too much preoccupied with technical improvements of the plant and have somewhat ignored the men who ultimately have to run the plants and who must be in a right frame of mind if maximum advantage is to be secured from the available physical resources and plant. The McGraw-Hill publishing house has brought out a series of useful books,—the Industrial Organization and Management Series", dealing with this aspect of industry.

The approach to this subject in the present book is practical. It deals section by section with topics like: "Adjusting the Human Resources", "Developing Attitudes and Sentiments", "Using and Abusing Incentives", "Bargaining with Individuals and Groups", "Mobilizing the Brainpower of an Organization" and "the Ways of Executives and Supervisors". In each section a number of practical cases are taken up and topics for discussion arising therefrom are suggested and certain principles are derived. It may be that conditions in the Western countries are different and, therefore, the psychological background may have but a remote bearing to the conditions existing in our country. An intelligent person will, however, by studying particular cases, easily grasp the principles underlying their studies and apply the principles *mutatis mutandis* to conditions existing in our own country. When one considers the loss of industrial resources as a result of strikes, slackness of workers, disorganization, lack of co-ordination of work in a factory and other human elements, one cannot under-estimate the importance of a concerted effort to grasp the principles that should determine the relations between the

executive and the worker in an industrial organization and the principles that make for maximum output by a harmonious working of the labour force. But understanding of principles is not enough. The executive should be trained to apply these principles in actual life. I feel that books like the present are helpful in achieving both the objectives.

K. R. F. KHILANI

**Heat**, by Archie G. Worthing & David Halliday (John Wiley & Sons Inc., New York; Asia Publishing House, Bombay), 1948, pp. xii+522. Price \$6.00.

THE BOOK OPENS WITH A CHAPTER ON SOME laboratory procedures — methods of observation; record and treatment of data; presentation of a report; etc., etc., — and sets the pattern on which the whole book is cast. It is specially written for the benefit of those who are taking up a serious study of heat for the first time, and the authors have, therefore, been at pains to emphasize the physical and the experimental sides. The whole book abounds with neatly drawn diagrams, sketches, charts, graphs and tables of constants. Numerous applications of the principles to industry and every-day life are discussed and will be greatly appreciated by young readers as aids to a clear understanding of the subject.

Each chapter is preceded by an introduction giving the relative importance of the topics discussed. After every discussion examples have been worked out to elucidate the manner of calculating the results from the data provided; in many cases alternative methods for the determination of a quantity are given. Wherever possible methods and results of recent developments have been incorporated. As an exercise to the student, a large number of carefully selected problems are added at the conclusion of each chapter. In general the treatment is elementary but complete within limits, and the derivation of formulae which require the use of advanced mathematics or understanding of complicated physical processes is left out. For the benefit of the diligent student who might like to go deeper, the derivation of Maxwell law of distribution of velocities is given in the appendix. Short biographies of great personalities in physics mentioned in the text form a most interesting feature of the book.

The topics covered in the book are generally those prescribed for pass B.Sc. course

in Heat of Indian universities and the book will be greatly welcomed by a large body of undergraduate students. The treatment is in some ways different from what the Indian students have been accustomed to, but the departure is all to be desired and will be found most refreshing and stimulating. It is a modern text-book in every way. Considering the scope of the book the authors, as experienced teachers, have done a service to the student world by presenting the material in such an attractive form. There is perhaps nothing the reviewer would like to say by way of criticism, but he feels that the chapter on "Kinetic Theory" might have been written in slightly greater detail. Also, a knowledge of the properties of substances at low temperatures has become important both in science and in industry. A discussion of the liquefaction of gases and other methods of producing low temperatures could have been included with profit.

The get-up is excellent and the book is strongly recommended for adoption by Indian universities.

P. K. K.

**Chemical and Electro-plated Finishes: The Protective Treatment of Metals**, by H. Silman (Chapman & Hall Ltd., London), 1948, pp. xiii+414. Price 30s. net.

INTENSIVE AND EXTENSIVE SCIENTIFIC investigations into the principles underlying the processes of electrochemical and purely chemical methods of supplying protective and decorative coatings on metals have led to vast advances in the industrial techniques employed in these fields. Mr. Silman has presented in this book a very readable account of the subject which will be welcomed at once by those interested in the underlying scientific fundamentals, and those who are mainly interested in the technical applications.

Chapters I to IV deal with the preparation of the metal surfaces for the various finishing processes. All the methods are dealt with in sufficient detail, and the data on the plants and machinery supported by choice illustrations enable the interested readers to benefit greatly by the information given. The recent developments in electrolytic pickling are included and also the electrolytic methods of polishing of metals. The latter subject has roused considerable



interest in recent years both on account of the reduction in cost and in labour, and also because of the fact that in some instances, as in the case of aluminium, it is possible to produce a higher degree of finish than is obtainable by mechanical methods of polishing. There can be little doubt that electrolytic methods of polishing have a considerable future. The electro-polished surface is usually free from strain while the technique enables a variety of surface finishes ranging from a full bright to satin or matt finishes.

Chapter V gives a very helpful account of the various methods of metal colouring that are now successfully adopted on a large scale, processes such as phosphatizing, in which metal surfaces are suitably treated with solutions of chemicals to develop a coating which has a good degree of rust resistance and other desirable properties. The recipes commonly used are given in fair number, with the compositions in the oz.-gal. or gr.-lit. system according, perhaps, to the available data.

Chapters VI to XII covering nearly 160 pages of the book are devoted to the details of the modern electro-plating methods and plants. The hardness, uniformity, and the durability of even thin coatings are the chief merits of this important finishing process which has now been developed into an art well based on a growing science. The modern methods of bright nickel plating have received special attention over nearly 12 pages.

Chapter XIII deals with the important subject of finishing of light alloys of aluminium and magnesium. Here a good account is given of the industrial methods of anodizing and subsequent finishing.

The last chapter, XIV, bespeaks the scientific character of this apparently professional book, and gives the various standard testing methods for quality control.

The book cannot claim to be a complete guide to the processes described and does not claim to be so. It should, however, meet adequately the requirements of designers and engineers and the processing technologists. There are also select references to original literature at the end of each chapter. The book is well printed with many clear photo reproductions and neatly arranged with an appendix of useful reference tables and indexes. The book is highly commended.

M. A. GOVINDA RAU

**Standard Design of Reinforced Concrete Road Bridges**, by L. E. Hunter (Chapman & Hall Ltd., London), 1948, pp. xv+176. Price 25s. net.

STANDARDIZATION IN DESIGN AND CONSTRUCTION of reinforced concrete road bridges is of the highest importance, especially in the present "post-war" period when damaged communication systems have to be set right without much delay to bolster up the shattered economy of many countries. The stereotyped methods of design adopted in the pre-war period were uneconomical and caused considerable loss of time in the execution of much-needed bridge projects. This book has been published at the most appropriate time, and it is certain to be of invaluable assistance to students, bridge designers and practising engineers. The author has to be congratulated on his fine effort in publishing so useful a book on an extremely difficult subject.

The author has aptly stated in the preface that it would take normally a life-time or more to deal adequately with the subject. It is indeed remarkable that he has so dexterously compressed a vast amount of useful information in a single volume of 176 pages.

The book deals with simplified designs on reinforced concrete road bridges of the various types generally used. It includes also one chapter on steel bridges. It abounds in a large number of valuable sketches, design tables and diagrams which, along with the formulae and calculations used to arrive at the dimensions of various members comprising a bridge, will definitely simplify the work of bridge engineers in the evolution of sound and economical designs.

This book should prove particularly valuable for students in engineering colleges of Indian universities.

R. K. N. IVENGAR

**The Indian Cotton Textile Industry — 1947-48 Annual**, by M. P. Gandhi (Gandhi & Co., Bombay), 1948, Vol. XI, pp. xiv+184+xxvii. Price Rs. 6.

MR. M. P. GANDHI DESERVES TO BE COM-  
plimented for bringing his 1947-48 annual of the Indian Cotton Textile Industry which, in a comprehensive and well-arranged manner, brings to light all the important developments of the cotton textile industry in India during 1947-48. The statistical

information which was not available in earlier annuals have been mostly made good and it is hoped that in future annuals more complete statistical data would be included.

The contents of the annual have been very ably classified and should serve as a very valuable ready reference book worthy of the library of all those interested in the textile industry, — students, economists, industrialists and business men. While chronologically detailing the various developments that lead to the discontinuance and reintroduction of the cotton control measures regarding output, price and distribution, he has analysed and given certain very valuable suggestions. So long as the Government of India depend for their advice and guidance mostly on those engaged and actively interested directly in the profits of the industry, and as long as they do not have on their staff specially qualified and able men in the Textile Directorate, no continued and useful policy regarding the textile industry can be evolved. In the case of such an important national industry like the cotton textile industry a policy of make-shift just to meet the exigencies of times is adopted which, in my opinion, gravely affects the future of the textile industry. There are quite competent men well able to determine and advise the Government but may not be able to do so as long as their loyalties remain divided between his employer on the one hand and the Government who seek his advice from time to time without a chance to put such advice to practise on the other. Both on account of its natural importance and on account of the varied and several most complicated problems involved at each phase of the cotton textile industry, the Government of India should examine the advisability of having a textile directorate manned with competent and well-qualified staff specially trained for the purpose on a permanent basis.

The several important problems that face the Indian cotton industry both now and in future have been ably discussed by Mr. Gandhi and deserve careful reflection by all concerned in the development of the industry in India.

B. K. MURTHY

**Microwave Transmission Circuits**, edited by G. L. Ragan (McGraw-Hill Book Co. Inc., New York), 1948, pp. xvii+725. Price \$8.50.

THIS VOLUME, WHICH IS NO. 9 OF THE "Radiation Laboratory Series", deals with the theory and practice of coaxial cables and wave-guides used in the propagation of microwaves. Seven authors who worked in the M.I.T. Radiation Laboratory have contributed to it under the heads: Elementary Line Theory, Materials and Constructional Techniques, Rigid Transmission Lines, Flexible Coupling Units and Lines, Transition Units (between coaxials and wave-guides), Motional Joints, Tuners, Power Dividers and Switches, Theory of Microwave Filters, and Design of Microwave Filters.

Although the earlier volume, No. 8, of the Series entitled "Principles of Microwave Circuits" covers the theoretical aspect of the subject, a certain amount of repetition has become inevitable in the present volume for the sake of completeness. The chapter on Theory of Microwave Filters covers aspects applicable to low frequency waves also and it is followed by a chapter dealing comprehensively with the design of microwave filters. "The theory and techniques described in these two chapters", it has been stated, "came too late to play a significant rôle in the war, but they should prove extremely useful in peace-time applications."

The term "microwave" is as vague as ever. For example, Vol. 11 on Microwave Measurements defines microwaves as extending from 1 mc./sec. to 30,000 mc./sec. In Vol. 8 the word microwave does not necessarily imply a particular range of frequencies, but "a characteristic technique and aperture field". In the present volume the term "microwave" may be assumed to include all frequencies greater than 1,000 mc./sec., but it has been specifically stated that the circuits described operate in the range from 2,500 to 25,000 mc./sec. In conformity with other volumes of the series *mks* units are used throughout the book; the text matter is well illustrated. The volume is beautifully got up, and it is a monumental contribution to the rapidly growing literature on microwaves.

B. N. SINGH

# NOTES & NEWS

## Precision Graduation of Scientific Glassware

MR. H. DUTTA, CHIEF GLASS Technologist, *Ganga Glass Works Ltd.*, Bulwali, writes as follows :

"Students of science and technology and research workers, all concerned with measurements of volume, demand accurately graduated and properly standardized measuring vessels for their work. There are a few firms in India turning out graduated glassware, but the bulk of the articles turned out have not attained the accuracy of the N.P.L. (*National Physical Laboratory*, Teddington) 'A' or 'B' grade. It would be realized that the expense and labour spent on accurate or faulty graduation are nearly the same, and with a little extra care it should not be difficult to turn out graduated ware of dependable accuracy in India."

The writer of this note recently visited U.K. to study the methods of graduation and calibration employed by manufacturers of scientific glassware in that country. The following notes based on his experience and study may be of interest to Indian manufacturers :

1. *Chemical Composition of Glass* — No definite composition of glass has been prescribed for scientific ware. The coefficient of cubical expansion determines the choice of glass employed for volumetric vessels. As the coefficient of cubical expansion of water is much higher than that of glass, it would be sufficient for most purposes to use low-alkali high-lime silica glass for making measuring vessels. Pyrex glass is generally employed wherever high accuracy is demanded.

2. *Method of Graduation* — The methods for marking vessels which are required to be accurate for contents, e.g. cylinders, flasks, and those which are required to be accurate for delivery, e.g. burettes, pipettes, are too well known. These methods which have been practised for some decades still prevail for precision jobs and for small production. For mass production of items, e.g. measuring glasses, the method is to employ pre-graduated paper transfer scales. After applying the trans-

fers, the articles are passed through a furnace when the graduation is permanently transferred to the glass.

The major difficulty in adopting this method in India is that the transfer scales have to be imported and they can be employed only if the sizes of the articles conform accurately in every respect to the standard. Any variation, however slight, would render their use out of question. More recently an ingenious printing machine and a new technique, "Silk Screen Printing", have been developed in Britain, which will render possible the precision graduation of syringes, ampoules, measuring glasses, etc. Although not directly applicable to all types of scientific glassware, these developments have great possibilities in India, and their adoption demands close consideration.

The scientific associations in India have to examine the position connected with the development of precision instruments required for research, education and professional work, and co-operate with manufacturers in their production. Standards have to be evolved and prescribed for adoption in India. It should be made incumbent on manufacturers to stamp their names on articles, and a certifying agency as in the U.K. should be established to issue certificates of accuracy for scientific glassware.

## Synthetic Glycerol

THE FIRST SYNTHETIC GLYCEROL plant, constructed by the *Shell Chemical Corporation* at Houston, Texas, went into operation last September. Commercial success has been attained for the first time since the discovery by Scheele, 170 years ago, that glycerol can be produced synthetically (*Chem. Age*, 1948, 59, 584). Employed in the manufacture of high explosives, surface coatings, and some 1,500 other applications, glycerol has become a heavy chemical in its own right, whose availability as a by-product of soap manufacture is no longer looked upon as a satisfactory sole source of supply.

The starting material is propylene produced from petroleum

hydrocarbons. Chlorination of propylene yields allyl chloride and hydrolysis of the chloride to allyl alcohol which in turn is chlorohydrinated are the principal stages in the process. The resulting glycerol dichlorohydrin is hydrolysed to glycerol.

98 per cent pure propylene is employed in the process. A suitable excess of propylene is maintained, the mole ratio of propylene to chlorine being about 7 : 1. A temperature of 500° to 580°C. is maintained during the reaction and yields of allyl chloride as high as 85 per cent are reported. The reaction products are first freed of hydrogen chloride by water scrubbing. The organic chlorides are removed by a kerosene wash and are then separated by fractionation. The residual propylene is recycled to the chlorination reactors, which it enters preheated to 400°C. by the outgoing products of reaction.

The hydrolysis of allyl chloride to allyl alcohol is carried out continuously at 150°-160°C. A pressure of about 200 lb./sq. in. is necessary to maintain a liquid phase. The allyl chloride concentration in the aqueous solution is of the greatest importance and the optimum concentration maintained is about 1.25 N., i.e. corresponding to about 5 per cent sodium hydroxide as the hydrolysing medium. Another important factor in the hydrolysis reaction is the alkalinity of the solution. Using sodium carbonate instead of sodium hydroxide, the ether formation is successfully repressed well below 10 per cent. However, the evolution of carbon dioxide results in undesirably high pressure. Only a small amount of sodium carbonate is, therefore, employed and the proper pH is restored by successive additions of caustic soda to keep carbon dioxide in solution. An alcohol yield of over 90 per cent is obtained. The allyl alcohol is readily recovered in its azeotropic (71 per cent) composition by simple stripping.

The hydrolysis reactors must be carefully designed to prevent allyl chloride accumulating in overhead pockets and causing corrosion.

The allyl alcohol is readily chlorohydrinated in dilute solution by contact with gaseous chlorine. The reaction temperature must be maintained at 20°-25°C. by removing the heat of reaction. Starting with a 5 per cent solution of alcohol in water, 92 per cent yields of

the chlorohydrinated allyl alcohol can be readily obtained. The product of this reaction is hydrolysed without purification. Aqueous alkali containing 10 per cent caustic soda and 1 per cent sodium carbonate gives the best results. Preferred operating temperature is 150°C. with a contact time of 30 minutes. About 95 per cent yield can be attained in the hydrolysis.

The crude glycerol is concentrated to 85 per cent in flash evaporators. Further purification to 99 per cent is effected by a combination of solvent extraction (with xylene) and vacuum fractionation.

In the alternate and equally efficient method, also successfully worked out by *Shell*, the allyl chloride is directly chlorohydrinated. By close control of the pH and avoiding the presence of gaseous chlorine in the system, it has been possible to obtain yields of glycerol dichlorohydrin in excess of 95 per cent. Glycerol chlorohydrin is hydrolysed partially (50 per cent) by treatment with lime in a stripping column. The resulting glycerol epichlorohydrin is distilled overhead and is readily hydrolysed to glycerol by treatment with caustic soda and soda ash. This process results in a saving of nearly one-half of the caustic soda consumption by the substitution of lime.

The glycerol manufactured by the *Shell Chemical Corporation* meets the very stringent specifications of *U.S. Pharmacopoeia*. The product is water-white, odourless, and assays more than 90 per cent glycerol.

### Lime-zeolites for Water Treatment

THE ADVANTAGES OF THE LIME-zeolite process for water treatment are being increasingly recognized (*Chem. & Ind.*, 1948, No. 44, p. 695). The use of carbonaceous zeolites for softening municipal and industrial water supplies in conjunction with lime, overcomes many of the disadvantages encountered in the lime and lime-soda methods. The carbonaceous zeolites, as ion-exchange materials, are immune to acid attack and are equally effective as the synthetic calcium-alumino-silicates in eliminating supersaturation due to calcium and magnesium salts and in absorbing small amounts of excess calcium hydroxide. They can be regenerated with acids.

The treatment is conducted in 3 stages. The first stage includes

treatment of water with excess of lime adding, if necessary, a coagulant such as activated sodium silicate, alone or in conjunction with aluminium sulphate. The effluent at this stage should analyse to the following: alkalinity to phenolphthalein, 40-50 parts  $\text{CaCO}_3$  per million; alkalinity to methyl orange, 60-70 parts  $\text{CaCO}_3$  per million; hardness, 60-70 parts  $\text{CaCO}_3$  per million + the permanent hardness present in the untreated waters. It is not necessary to filter the water leaving the lime-treatment plant, as any suspended matter will be retained in the next stage.

The effluent is passed in the second stage through a pressure filter containing a stabilizing bed of carbonaceous zeolite in the calcium form. The carbonaceous calcium zeolite acts both as a catalytic and mechanical filter. It removes suspended matter, completes the lime-bicarbonate reaction, retains the precipitated calcic carbonate and magnesium hydroxide and absorbs any excess of lime. Tests on this stabilized effluent should be approximately as follows: alkalinity to phenolphthalein, nil; Alkalinity to methyl orange, 10-15 parts of  $\text{CaCO}_3$  per million; hardness, 10-15 parts of  $\text{CaCO}_3$  per million + the original permanent hardness in the untreated water, and pH 6.9-7.1.

The accumulation of calcium carbonate and magnesium hydroxide is periodically removed from the stabilizing bed by means of acid.

In the third stage the effluent from the stabilizing plant (suitable without further purification for many purposes) is passed through a normal base-exchange softener which may contain a zeolite of the natural sodium-alumino-silicate or carbonaceous type, in which the sulphates, chlorides and nitrates of calcium and magnesium are converted into the corresponding sodium salts.

The effluent from the base-exchange unit is ideal for most industrial purposes and should be of the following quality: alkalinity to phenolphthalein, nil; alkalinity to methyl orange, 10-15 parts of  $\text{CaCO}_3$  per million; total hardness, nil; pH 7.0-7.2, and free carbon dioxide 2 p.p.m.

The zeolite is regenerated by treating the bed in an upward direction with a solution of sulphuric (0.5 per cent) or hydrochloric acid (up to 10 per cent). The base-exchange unit is regene-

rated with sodium chloride in the conventional manner.

### Manufacture of Urea

THE THEORETICAL ASPECTS OF urea synthesis and the methods employed in its industrial production have been recently described (*Chem. & Ind.*, 1948, 60, 22).

The two fundamental steps in the synthesis starting from the relatively cheap raw materials, ammonia and carbon dioxide, are the formation of ammonium carbamate and the dehydration of carbamate into urea. The reaction being an equilibrium reaction, the rate of transformation of ammonia into urea in a single operation never exceeds 50 per cent. The recovery of residual ammonia and carbon dioxide becomes incumbent for economic production. Where gases are utilized in an auxiliary production, such as preparation of ammonium sulphate by the gypsum process, the recovery is relatively simple. Ammonia and carbon dioxide are compressed into an autoclave (min. pressure, 120-170 atm.) at 170° to 180°C. The transformation into carbamate, and its dehydration into urea is complete in 15 to 20 min. The reaction products enter a distilling column in which the pressure is reduced to 2 to 3 atm. and the temperature kept at 75° to 80°C. Unreacted ammonia and carbon dioxide are led out from the top of the column to the auxiliary plant. Urea is evacuated from the bottom in the form of a concentrated solution and crystallized. The heat of reaction serves to pre-heat the gases and maintain the temperature of the autoclave at the desired level.

The reactants must be pure. Corrosion of equipment is a serious problem, the outlet valves of the autoclave being the most vulnerable. A special alloy has been elaborated to overcome corrosion.

The gaseous products containing about 6 per cent of water vapour, escaping from the top of the distilling column, are recycled in the absence of an auxiliary plant. This proportion of water vapour is admissible. The compression of the gaseous mixture is not easy, and it is inconvenient to introduce them under pressure into the autoclave in a continuous manner. The gases may be introduced in the form of aqueous solutions pumped continuously into the autoclave under pressure and supplemented with additional quantities of gases. After the

reaction, the mixture is submitted to a double distillation. In the first distillation, carried out under pressure, the major portion of ammonia is liberated. It is liquefied and recycled. After the second distillation, at atmospheric pressure, the liberated gases are dissolved in a minimum quantity of water and recycled into the autoclave. This process is adopted by *Du Pont de Nemours* because of its several advantages.

The A.F.C. process operated in France employs a colloidal suspension of ammonium carbamate in a mineral oil medium. By mixing carbon dioxide and ammonia in a paraffin medium, suspensions containing 50 per cent or even more of ammonium carbamate are obtained. These are capable of being pumped without difficulty. The suspension is introduced under pressure continuously into the autoclave at the same time as fresh gases. After the reaction the oil is separated by decantation. The advantages are the non-formation of crusts by ammonium carbamate, and the easy dissipation of heat.

After entering the hot autoclave the carbamate melts or dissolves in the reaction mixture. If the reactants enter from the bottom, the oil rises to the top where it is constantly evacuated. The autoclave is always full of carbamate. The theoretical heat balance is nil or slightly positive starting from a rate of transformation into urea of 40 per cent, if ammonia is introduced above its critical temperature, and the suspension contains 50 per cent of carbamate. Urea solution is drawn from the bottom of the attached distilling column, concentrated under vacuum and crystallized. The gaseous mixture from the top is recycled to produce the suspension.

K. VYASULU

### The Pneumatic Balance

THE PRINCIPLE OF THE PNEUMATIC balance and some of its applications have been recently described (*Ind. Chemist*, 1948, 214, 676). A number of devices based on the principle of the Electroflo Pneumatic Transmitter developed by the *Electroflo Meters Co. Ltd.* have been given with special reference to their applications in industry.

In the pneumatic balance the pan and the measuring weights of a conventional chemical balance are replaced by a diaphragm which is arranged so that an air pressure applied to it can be

regulated just to balance the material which is being weighed. If a baffle is attached to the indicating pointer of the balance, and a nozzle supplied with a constant source of air is fixed beneath it, the distance of the baffle from the nozzle will control the air pressure at the nozzle. This pressure can then be used to indicate on a gauge when the correct pressure is applied on the diaphragm to balance the material weighed. Such an arrangement will give a degree of precision comparable with the chemical balance, as a minute movement of the baffle will produce a big deflection of the gauge connected to the nozzle. By applying the nozzle pressure direct to the balancing diaphragm the system becomes self-balancing. Great sensitivity is attainable without any detectable hysteresis as there is absence of movement.

The obvious advantages of pneumatic balance are for those applications where it would be necessary to run connecting pipes containing corrosive, inflammable or toxic fluids into control rooms, etc., or employing electricity where there are fire and explosion hazards. The pressures and flows of corrosive fluids can be measured and transmitted by this means employing a diaphragm chamber and diaphragm constructed of suitably resistant materials, thus eliminating the need for liquid seals and purging systems in the majority of cases. The measurement of viscous fluids such as heavy fuel oil and pitch has been successfully undertaken; the only precaution being that the measuring chamber and the connecting lines are kept at the same temperature as the main feed line. The method may also be successfully employed with liquid ammonia and other fluids which evaporate at normal temperatures.

The pneumatic balance principle can be employed to convert into measurable air pressure any quantity or condition which can be caused to apply a force to the transmitter weigh-beam, and this pressure may be used not only for indicating or recording, but also for controlling the force. One application is, for example, that of the level measurement and control. Another adaptation of this principle is the detection of small force changes in a system where differences in vapour pressures of 2 fluids are to be determined. A device based on this principle has been perfected to

detect the presence of  $\frac{3}{10}$  of 1 per cent of butane in isobutane.

Many of the difficulties usually encountered with orthodox flow meters may be successfully overcome by using the force balance method employing the same principle. Owing to the absence of displacement, the flows of condensable vapours such as steam may be measured without the necessity for constant-level reservoirs. Other applications in which the principle has found use are: measurement and control of speed of rotation, the pull of a speed governor being applied to the beam or of torque, weight, etc.

### Grease for Rust Prevention

IN THE TROPICALIZATION OF MOTOR vehicles it was found necessary to devise a means for protecting the threads of nuts and bolts against rust, and to allow for their easy removal. A number of rust inhibitors were tried and an aluminium stearate grease composition was selected as satisfactory (*Can. J. Res.*, 1948, 26F, 347). The grease contains: 2 per cent zinc chromate (as used in paint formulation), 5 per cent graphite (fine flake), 7 per cent aluminium stearate and 0.2 per cent stearic acid; the remainder was acid and clay treated Columbia Distillate having a viscosity of approximately 90 S.U.S. at 210°F. The graphite and zinc chromate were slurried with some of the oil and passed twice through a colloid mill or other shearing device to disperse them. The remainder of the oil and other ingredients were then added and, after mixing in the cold, the dispersion was heated to 160°C. with constant stirring. This was then allowed to cool and let stand for 24 hr. before use.

Plates with bolts and nuts after treatment with the grease composition were subjected to immersion in sea water for 8 hr. a day; they were then heated in water at 50°C. and after 1 hr. the heat was cut off and the atmosphere allowed to cool and condense slowly. This cycle was repeated every 24 hr. After 10 weeks under these severe conditions of test it was found that the bolts and nuts treated with the special grease composition could be loosened by hand and easily turned.

### New Uranium Mineral

A NEW RADIOACTIVE MINERAL containing uranium has been

reported from Belgian Congo by Dr. Paul F. Kerr of the Columbia University. The mineral was discovered by Dr. J. F. Vaes of *Union Minière*, Jadotville, Belgian Congo. Laboratory tests show that the material is an unknown uranium mineral. It has been named "Sengierite" in honour of Edgard Sengier who directed wartime mineral production in Belgian Congo.

Sengierite is found in small green crystals which cling to a chlorite-talc rock found in the mines. It is similar to the American uranium mineral, carnotite, except that sengierite is a copper uranium mineral while carnotite is a potassium uranium mineral (*Sci. News Letter*, Oct. 1948, p. 248).

### Castor Oil in Steel-casting

CASTOR OIL PROMISES TO BE AN effective substitute for linseed oil in steel-casting as a binding material for sand cores. The quantity required is usually 2 per cent on the weight of sand employed. At a conference called at Leamington Spa by the Steel Castings Division of the *Iron & Steel Research Association*, it was reported that castor oil may be effectively substituted for linseed oil (*Chem. Age*, 1948, 59, 630).

### New Instruments for Textile-testing

EXHIBITED AT THE 19TH ANNUAL meeting of the *Textile Research Institute Inc.*, U.S.A., held in New York City last November, was a new tensile tester which incorporates new electronic principles of load-weighing and which provides full-scale load ranges from 2 gm. to 1,000 lb. In another instrument an acoustic pulse is introduced into the material being tested and its time of propagation is measured between a transmitter and a receiver. By this means the elastic modulus of the material is determined with great simplicity and speed. Electrical instruments for moisture determination and control as well as measuring instruments for accurately determining the diameters of yarns were on exhibition. Another instrument was a non-contact continuously indicating measuring device for the diameter of textile yarns and fibres.

Other instruments on display were a high-speed microtome for cutting extremely thin sections of textile materials for microscopical examination, and a knit fabric shrinkage tester.

### Livestock Feed from Citrus Wastes

ONE OF THE MOST INTERESTING developments in the citrus fruit-processing industry in U.S.A. has been the development of valuable by-products which eliminate the high cost of citrus waste disposal. Waste pulp and peelings are converted into high-grade shredded livestock feed. It contains large percentages of carbohydrates and substantial quantities of fat and minerals. The liquor which drains from pulp and peel as it passes the livestock feed processing can be converted into valuable citrus molasses by vacuum evaporation and added to mixed cattle feeds as a substitute for more expensive sugarcane molasses. The juices can also be converted into industrial or beverage alcohol. Oils are used as bases for flavourings, beverage bases, perfumes and in the preparation of certain pharmaceuticals. Citrus seeds yield oils useful in the manufacture of sulphonated oils. 60 per cent of the pectin of commerce is obtained from waste orange, lemon and grape-fruit peel.

Other possibilities are: ascorbic and citric acids can be obtained from the waste; sugars can be utilized in the production of food yeast; enzymes useful in the early processing of fruit juices can be prepared. — U.S.I.S.

### Photo-typesetting Machine

AT THE RECENTLY OPENED LABORATORIES of the *Printing Research Association* at Leatherhead, Surrey, equipment has been installed to demonstrate a system of photo-typesetting which dispenses with cast metal type. Its trade name is "Rotofoto".

The new method is a purely photographic process which should be useful in printing type by the lithography and gravure processes now widely used in the production of books and periodicals. The new system appears to be as cheap as standard printing practice; and it has the obvious advantage that the photographic master-copy of a book's typescript, from which reprints can be made, can be stored in a small space.

The "Rotofoto" equipment comprises a Monotype keyboard, a line-projector, and a make-up machine for producing paper proofs and film transparencies. A punched paper record, representing the "copy", is first made by a Monotype keyboard operator. This paper record is transferred to the line-projector which contains the mechanism of the Monotype composition caster with the main difference that a photographic negative takes the place of the matrices.

The letters punched in the record are photographed on a roll

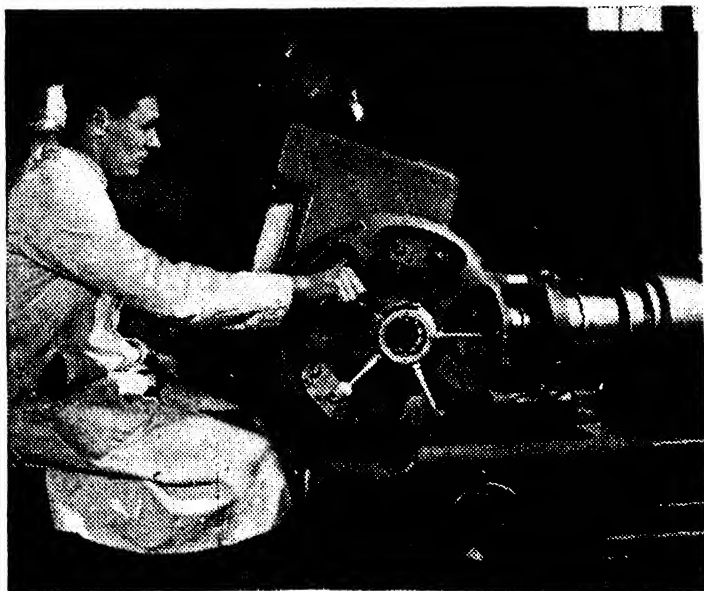


PHOTO-TYPESETTING MACHINE.

of 35 mm. cinematograph film carried in a camera under the type negative. After a full line has been completed the film is advanced, the distance depending on the space required between the lines, and the next line is photographed. The line-projector operates at about the same speed as a Monotype casting machine.

Readers' and authors' corrections are dealt with separately and contained in other rolls of films: the make-up machine operator introduces the corrected lines during the process of making a perfect photographic transparency of the type matter.

The operations are simple and rapid and produce transparencies of high fidelity. Any type-face can be employed by using the appropriate negative, the type sizes can be controlled by the magnification employed in the make-up machine. At present 3 negatives are used to cover the complete range of composition sizes.

### Mica Substitutes

GERMANY PRODUCED, DURING THE World War II, large sheets of mica using natural mica available in powder form (usually a waste product in mica industry). The mica powder was melted in graphite crucibles at 2,350°F. and cooled under regulated conditions in the complete absence of any vibration. During cooling a temperature gradient and a horizontal magnetic field are imposed. This process of forced formation of crystals in large laminated sheets was developed in the *Kaiser Wilhelm Institute for Silicate Research*.

Various substitutes for mica were developed in Germany during the war. They were mainly compressed paper products impregnated with artificial resins. The more important of these were: "Pertinax" developed for high-tension condensers, "Paxolin" and "Formalite", two phenol-formaldehyde compositions, and "Leatheroid", a fibrous material produced from cellulose. None of them seriously threatened the natural mica market.

The Massachusetts Institute of Technology has announced (*U.S. Tariff Commission Report, No. 130*) recently the development of inorganic films closely resembling mica in their important electrical and physical properties. When certain highly hydrated gels such as, for example, bentonite are suspended in water, and substan-

tially monodisperse fractions of the colloidal particles are separated out, the gels of these fractions form, upon drying, coherent, self-supporting and flexible films. No binder is necessary. When these films are heated under pressure their strength and transparency are greatly increased and their electrical resistance is comparable to that of mica. Although relatively polydisperse fractions also form films, those made from substantially monodisperse fractions are of superior quality. Monodisperse fractions may be obtained by allowing suspensions of clay to separate into layers according to size by gravity, and later to separate them in a super-centrifuge.

Such films, when made from bentonite (hydrated aluminium or magnesium silicates), are very similar in chemical composition to mica and when dried to a low-water content, exhibit many of the properties of mica. Films of different thicknesses can be made, depending upon the thickness of the original layer of gel. Similarly, a laminated product may be made from a number of thin sheets.

Intensive research is under way to obtain more information regarding the characteristics of the films under a variety of conditions, and in order to develop more fully the theory of their formation. Careful consideration is being given to the suitability of various treated films as substitutes for natural mica in condensers, radio tubes and other applications.

An arrangement has been made by the *Institute* with the *Research Corporation* of New York to undertake the commercial development of the patented process.

Colloidal clays are inexpensive and widely distributed. The quantities required for synthetic mica sheet production are relatively insignificant. The process would yield films or thick sheets as desired, which are technically perfect in physical structure and chemical analysis. The possible substitution of synthetic sheets for the built-up mica now universally used in electrical designs, appears to be the most significant economic aspect of the synthetic development.

D. B. SAHANA

### Sterols from Wool Fat

A RECENT PATENT TAKEN OUT BY a Netherlands worsted yarn-spinning mill relates to an improved method for separating sterols, e.g. cholesterol, from the liquors ob-

tained in wool-washing. The sterols may subsequently serve as basic materials for the manufacture of vitamin D and some hormone preparations.

The unsaponifiable portion of wool fat is heated in the presence of a metal salt, e.g. anhydrous calcium chloride, the resulting addition product is treated with a solvent, e.g. acetone, whereby the addition product is partially or entirely dissolved. The method, it is claimed, is economical and gives a higher yield of sterols.

### Storage Life of Adhesives

THE STORAGE LIFE OF RESIN adhesives is an important criterion in their applications and usefulness. Studies on the storage life of "Tego-gluefilm" under tropical conditions are described in the *Indian Forest Leaflet No. 99*. The tests were conducted with veneers obtained from *Mangifera indica*, *Cedrela toona* and *Dalbergia sissoo*.

The studies reveal that the films when kept in an ordinary carton container, termed "open parcel", lose their power of adhesion in about 3 months. When packed in paper and then placed in an air-tight metal container, termed "sealed parcel", the films are usable up to about a year.

### Notes on Soldering

IN A NEW EDITION OF THE *Tin Research Institute's* handbook, *Notes on Soldering*, W. R. Lewis, reviews the recent researches on soldering process and presents a compilation of the more important facts which are likely to be of value in a large variety of industries.

Advances in soldering technique, with particular reference to mass-production methods of assembly, are discussed and the various forms of solder and methods of applying heat to the joints are described briefly so that their principles may be easily discerned and readily adapted for any particular application.

The principles underlying the production of "wiped joints" in lead pipes are fully covered and alternative types of joint in lead pipe are described and illustrated.

Aluminium, stainless steel, cast iron and other difficult-to-solder alloys are capable of being soldered by the special methods suggested.

Data on the behaviour of solders at various temperatures under tensile and shear stresses,



and under creep conditions, so important to the designer and to the production engineer, have been presented, together with notes on the properties and metallurgical constitution of the tin-lead solders.

The book also contains an adequate bibliography and a detailed scheme for the chemical analysis of tin-lead solders devised by Dr. J. W. Price.

This 88-page booklet, which is suitably illustrated with 47 photographs and diagrams, is available, free of charge, on application to the *Tin Research Institute*, Fraser Road, Greenford, Middlesex, England.

### Tars & Bitumens in Road Construction

ROAD RESEARCH TECHNICAL paper No. 12, *Properties of Road Tars & Asphaltic Bitumens in Relation to Construction*, published recently by the *D.S.I.R.*, London, discusses the properties of road tars and bitumens and their significance in terms of the behaviour of surfacings in which they are employed.

Bituminous and tar road-surfacings must resist the transient forces imposed by traffic, but yield to slower forces of expansion and contraction and they must retain these properties under conditions of severe exposure to weather. The properties of the binder such as viscosity, its capacity to adhere to aggregates and its resistance to deleterious effects of weather are discussed in the paper in relation to various types of road-surfacings.

The phenolic constituents of the tar are reported to give better adhesion properties than bitumen in the presence of water. Bitumens fluxed with tar oils have better adhesion than those fluxed with petroleum oils. It has been found that improved adhesion in pre-mixed surfacing materials can be obtained by the incorporation in the binders of surface active materials such as heavy metal soaps and sulphonated oils. In surface-dressing improved adhesion in wet weather can be obtained by treatment of the stone with a adhesion agents such as calcium oleate and iron oleate.

The measurement of brittle characteristics of tar and bitumens at low temperatures provide a useful means of following the deterioration in the binders caused by exposure to weather.

Road tars and bitumens are amenable to oxidation at atmospheric temperature, the effects being more detrimental to tar. Atmospheric oxidation, which is accelerated by light, is largely a dehydrogenation process leading to brittle resinous compounds. Removal of the phenolic constituents of tar reduces susceptibility to oxidation. The outstanding problem in this field is the development of an oxidation test for bituminous materials which will make it possible to predict road performance. Oxidation under pressure provides a reliable accelerated test.

### Building Research in U.K.

LOAD TESTS CARRIED OUT BY THE *Building Research Station*, *D.S.I.R.*, London, on full-scale structures which have been accepted as standards are described in the Special Report No. 1, published by the *H.M.S. Office*, London. Standard impact tests have been enumerated. Under strength requirements a factor of 2 has been suggested analogous to the factor of safety which allows for errors in assumptions, calculations and workmanship. Stiffness requirements prescribe that the structural part should recover appreciably after the test load is removed. The exact requirements under this head for each structural part are enumerated in a summary at the end of the report. Actual application of test loads and measurements of deflection or deformation are described including a number of photographs and a sketch to illustrate the testing of panels of a roof or floor. Vertical loads like dead load, superimposed load, impact load and lateral load (wind load mainly) are also dealt with.

The report is valuable to the designer, safety engineer, factory engineer and the industrialist interested in building design and construction.

*Clay-building Bricks* — This Bulletin, the first in the National Building Studies series, issued by the *D.S.I.R.*, London, contains the researches carried out at the *Building Research Station* on clay-building bricks.

The Bulletin is in 3 parts. The first part deals briefly with the manufacture of bricks from raw clay. The second describes the characteristic properties of brick such as compressive strength, water absorption and other properties including thermal conductivity, sound insulation, resis-

tance to weathering and the effect of soluble salts. Results of tests on the efficiency of brickwork are summarized in the last part. These notes are intended to indicate the limited applicability of the test data in actual practice.

The Bulletin stresses that in order to guarantee a satisfactory construction more attention should be paid to planning and design of building as a supplement to the test data.

References have been given to the relevant British Standard Specifications and the publications of the *Building Research Association*.

*Concreting in Cold Weather* — Measures which can be taken to enable concreting and bricklaying work to proceed normally in cold weather are described in the National Building Studies Bulletin No. 3, issued by the *Building Research Station*, *D.S.I.R.*, London. Precautions to ensure the continuation of work on buildings in cold weather chiefly relate to concreting and bricklaying, because (a) the water in mortar and concrete mixes freeze, and (b) such mixes harden more slowly at subnormal temperatures.

Among the steps that could be taken to allow concreting to continue under winter conditions, the use of calcium chloride in the mix is recommended to reduce the period of setting and hardening. The incorporation of calcium chloride (2 per cent on the weight of cement gives maximum efficiency) increases the rate of strength development, especially at the early stages and reduces the time-period during which it is necessary to protect the concrete. It also increases the rate at which heat is evolved from the cement during the early period of hardening. Another effect is to lower the freezing point of the wet mix. In order to obtain full benefits of the addition of calcium chloride use of insulation is recommended in order to conserve the heat liberated during setting.

Calcium chloride may be used with either ordinary or rapid hardening cements. It is pointed out that calcium chloride should not be used in reinforced concreting work where thin cover is allowed.

Special precautions recommended for urgent work when frost conditions are severe are: to pre-heat the materials and heating the framework and keeping the



newly placed concrete warm. The use of rapid hardening cement is recommended for concreting in cold weather.

Bricklaying is not influenced to the same degree as concrete-laying by winter weather. The composition of the mortar mix is varied; 2 parts of cement, 1 part of lime and 5-6 parts of sand gives good results. The use of calcium chloride in the mortar is not beneficial; mixing in a warm enclosure, pre-heating of water, keeping the mortar warm and insulation of the brickwork are the other precautions recommended to ensure work to proceed normally. The bricks employed should be kept dry and should not be dipped in water before laying but used in a dry condition allowing for the suction by adjusting the amount of mixing water in the mortar.

N. K. P.

### Rubber Seed Oil

EXPERIMENTS CONDUCTED BY THE Ceylon Government rubber technologist confirm the opinion expressed by the *Rubber Commission* that oil extracted from rubber seeds may form a suitable substitute for linseed oil. A ton of rubber seed oil fetches £150 and the extraction of oil on a large scale is regarded as a reasonable commercial proposition (*Chem. Age*, 1948, 59, 594).

On a conservative estimate, it is expected that Ceylon can produce about 10,000 to 12,000 tons of oil worth 10-12 million rupees. Ceylon may be able to export 3,000 tons of oil when the necessary plant for large-scale extraction is erected.

### Rubber Production Plan

A RS. 15 CRORE SCHEME FOR making India self-sufficient in rubber is envisaged by the *Indian Rubber Board* at its fourth annual meeting held at Bangalore. The *Board* suggested that steps should be taken for stepping up the country's rubber production of 16,000 tons to at least 40,000 tons within five years.

### World Rubber Production in 1948

THE TOTAL WORLD OUTPUT OF natural rubber during the first 9 months of 1948 by the Rubber Study Group of the F.A.O. was 1,140,460 metric tons, an increase of 213,360 metric tons over the comparable period in 1947. World stocks of rubber, excluding

stocks in Russia, are estimated at 812,800 metric tons, as on September 30, 1948, an increase of 36,474 metric tons since June 1948.

World production of 40,640 metric tons of synthetic rubber in September 1948 brought the total for 9 months to 403,860 metric tons and consumption for the year to date was 373,380 metric tons. Stocks of synthetic rubber increased from 70,740 metric tons at the beginning of the year to 114,300 metric tons at the end of September 1948.

### Mica Marketing Control Board

THE GOVERNMENT OF INDIA HAVE decided to appoint a Committee consisting of 15 members to consider and report on the practicability of setting up a Mica Marketing Control Board in India, and, if such a Board is considered feasible, to suggest its constitution and functions and also to work out a detailed scheme for the efficient marketing of mica.

Dr. M. S. Krishnan, Director, *Indian Bureau of Mines*, is the Chairman of the Committee. A representative of the Ministry of Commerce, one representative each of the Governments of Bihar, Madras, West Bengal and Rajasthan, and eight representatives of the trade are the other members of the Committee.

A meeting of the *Interim Mica Advisory Committee* was held at Madras on December 20 and 21 to consider, among other subjects, a scheme for mica-mining classes for training mine managers, levy of Mica Mines Labour Welfare Cess, railway priority for scrap mica and a revised rate of royalty for mica.

### Show Rooms for Forest Products

THE ESTABLISHMENT OF INDUSTRIAL show rooms at important centres in India, for the display of products utilizing timber and other forest produce on a commercial scale, is engaging the attention of the *Forest Research Institute*, Dehra Dun. Chambers of Commerce and Forest Departments of Provinces and States have been asked to make suggestions regarding the establishment and maintenance of the show rooms.

The purpose of the show rooms will be to bring out effectively the resources of the forests of the adjacent regions, to draw atten-

tion to established industrial uses for them and to indicate the scope for extending the utilization as revealed by research and development. The exhibits will be collected from Provincial and State Forest Departments, established industries utilizing forest products, and from the *Forest Research Institute*, Dehra Dun.

The questions likely to be examined separately for each centre will be whether the show rooms should form part of a general or industrial museum, whether it should be set up as an independent show room under Government management, or whether it should be attached to a recognized or large industrial undertaking.

### Exemption of Duty on Insecticides

AS AN EXPERIMENTAL MEASURE, the Government of India have decided to exempt from payment of customs duty insecticides, fungicides and weedicides, including the basic chemicals employed in their preparation. This exemption will remain in force for one year, after which the position will be reviewed.

To ensure that the materials imported are used for the purposes for which the concession is granted, importers are required to produce from manufacturers and agriculturists employing them, a certificate, within 6 months of importing them, to the effect that they have been properly utilized.

### The Indian Standards Institution

THE FIRST ANNUAL REPORT OF the *Indian Standards Institution (I.S.I.)* for the year ending 31st March 1948 shows an impressive development in the activities of the *Institution* in the short period of a year. The *Executive Committee*, in addition to dealing with the day-to-day administrative and organizational matters, has, at 4 meetings held during the year under review, made many important decisions relating to the basic structure of the *Institution* and its various Councils and Committees to deal with standardization in the national and international spheres.

The *I.S.I.* represents India on the *International Organization for Standardization* and new Secretariats for 2 Technical Committees for shellac and mica have been entrusted to the *I.S.I.* Accordingly, secretariats have been set

up and draft terms of reference for both have been prepared for circulation. The *Institution* has admitted 333 Sustaining Members and 23 Ordinary Members during the year. Invitation to neighbouring countries for Sustaining Membership of the *Institution* and collaboration in its activities were extended by the first President of the *I.S.I.*, Sri C. Rajagopalachari. The Government of Ceylon has already become a Sustaining Member.

The Textile Division Council of the *I.S.I.* held its first meeting on 7th August 1947, and appointed 6 Sectional Committees to deal with an approved list of subjects falling under the following groups: Textile standards (standard methods for testing textiles), cotton yarn and cloth, jute, wool; Textile chemistry; and Textile stores. Five of these, excepting Jute Committee, at their first meeting, appointed 22 Sub-Committees to deal with specific subjects.

The Textile Division Council has also been entrusted with the work of drawing up a specification for the Indian national flag in consultation with the Ministry of Defence. The specification is under preparation. Standardization of filter cloth for shellac is another subject under the active consideration of the *Institution*.

The Engineering Division Council at its first meeting, held during November 1947, appointed 13 Sectional Committees to deal with an approved list of technical subjects.

A report suggesting the adoption of the metric system of weights and measures was prepared by the Director and forwarded to the Ministry of Industry and Supply as directed by the *Executive Committee*.

The establishment of the Chemical Division Council has been expedited. Nearly 100 subjects for standardization have been proposed by the members and other bodies for consideration of the Division.

The formation of the Agricultural and Food Products Division Council and the Building Division Council has been postponed for the time being.

At the instance of the first President of the *I.S.I.*, Sri C. Rajagopalachari, the Director prepared a report on the rationalization of Indian coinage on the basis of the decimal system.

Close liaison has been maintained through *I.S.I.* between

India and National Standards Bodies of all countries, members of the *International Organization for Standardization* and the British Commonwealth of Nations. Information on standardization is being constantly exchanged between India and these countries. More than 170 references were received inviting opinions on draft specifications.

The sale of publications, in India, of foreign standard bodies, particularly those of the *British Standards Institution*, has been undertaken by the *I.S.I.*

A Sectional Committee on Quality Control and Industrial Statistics consisting of 18 members has been appointed, including Dr. W. A. Shewart, for the purpose of finalizing draft standards on quality control methods for India on the basis of existing standards. The Committee has since reprinted the *American Standards Association* Standard Z-1.3 on "Control Chart Method of Controlling Quality during Production", pending the formulation of an Indian Standard, on which a Sub-Committee is now working.

A nucleus of a library has been started. The present collection includes standards issued by various bodies in India, Great Britain, United States, Canada, Russia, Australia and a few other countries. During the period under review, 5412 specifications, 171 draft specifications and 89 proceedings of Committee meetings of various national organizations were received in the library.

#### A New Statistical Series

A NEW STATISTICAL SERIES, ENTITLED "Monthly Abstract of Statistics", showing the changing pattern of the economic activity in India, has been brought out by the Statistician to the Government of India in the Ministry of Commerce.

Figures are provided in the "Abstract" on population; employment; agriculture, industrial, mineral and power production; transport including civil aviation; value of contracts, foreign trade; banking, currency and finance; stocks and consumption of food, raw materials and manufactures; postal traffic; prices of precious metals, securities and commodities; cost of living; and industrial profits and disputes.

The statistics which have been collected from various sources are compiled and presented according to the International Standards.

The publication is priced annas 9 or 10d. per copy, and can be had from the *Manager of Publications*, Delhi.

#### Aeronautical Society of India

THE *Aeronautical Society of India* was inaugurated by Pandit Jawaharlal Nehru, Prime Minister of India, on December 27, 1948, at Bangalore. The *Society* (President, Mr. N. C. Ghosh, Director of *Civil Aviation* in India) has been formed for promoting the advancement of the profession of aeronautics. Its membership includes scientists, engineers and key-technical men of aviation organizations. The Prime Minister of India was elected Patron-in-Chief of the *Society*.

#### Electrical Communication Engineering Department — Indian Institute of Science, Bangalore

THE FOUNDATION-STONE OF THE new premises for the Department was laid by the Prime Minister of India on December 27, 1948, at the *Indian Institute of Science*, Bangalore.

The Government of India have made a grant of Rs. 4.25 lakhs for the construction of the building and equipment. In the design of the building, provision has been made for future expansion. The ground floor with a plinth area of 19,000 sq. ft. will be constructed for the present at an estimated cost of Rs. 2.75 lakhs. This will provide 4 big size laboratories and several smaller ones besides the lecture halls.

Instruction and research in electrical communication is being carried on at the *Institute* for the past 25 years as a part of electrical technology. The *Institute* was the first in India to introduce the subject as a separate study.

#### Crystallographic Exhibition

THE FIRST CRYSTALLOGRAPHIC exhibition in India was recently organized at Bangalore in connection with the 14th Annual Meeting of the *Indian Academy of Sciences*. The exhibits were arranged in the Physics Department of the Central College, Bangalore, and was visited by Pandit Jawaharlal Nehru, Prime Minister of India, who was on a visit to the city then.

Explaining the exhibits, Sir C. V. Raman, President of the *Indian Academy*, and the Founder-Director of the Raman Re-

search Institute, stated that unity of science was very well illustrated by the science of crystallography. The homogeneous nature of thousands of particles contained in the minerals could be seen there. Sir C. V. Raman, world's leading exponent of crystal physics, intends to start a museum of crystals with his fine collection as the nucleus. It may be mentioned here that the *Indian Academy of Sciences* has published, most appropriately, a symposium of papers on crystal physics to commemorate his sixtieth birthday which was recently celebrated in Bangalore.

### UNESCO Book

#### Coupon Scheme

**\$150,000 WORTH OF BOOK COUPONS** were delivered to representatives of 13 participating countries by the Acting Director-General, Gordon Menzies, during a special ceremony held during December 1948 at the *UNESCO* House, Paris. The scheme, devised to overcome foreign exchange difficulties will enable educational and scientific institutions of "soft" currency countries to buy publications from hard currency countries, while making payment in their own national currency. Coupons worth \$50,000 will be donated by *UNESCO* to Austria, China, Czechoslovakia, Greece, Hungary, Italy, Indonesia, Iran, the Philippines and Poland. The additional \$100,000 worth of coupons will be put on sale in China, Czechoslovakia, France, India, Poland and the United Kingdom.

A distributing body in each of the 13 countries will be in charge of allocating these coupons and will indicate the rate applicable for their purchase. The coupons can be used to buy books on education, science and culture

and can be used for subscription to periodicals or photo-copies.

Booksellers who accept these book coupons will be repaid by *UNESCO* in their respective national currencies.

### Announcements

*Indian Science Congress Session, 1950*: The 37th Session of the *Indian Science Congress* will be held at Poona from 2nd to 8th January 1950. Professor P. C. Mahalanobis, F.R.S., has been elected as the President of the Session.

The following have been elected Sectional Presidents: *Mathematics*: Prof. N. M. Basu (Aligarh); *Statistics*: Dr. P. V. Sukhatme (New Delhi); *Physics*: Dr. R. N. Ghosh (Calcutta); *Chemistry*: Dr. J. K. Chowdhury (Calcutta); *Geology and Geography*: Mr. J. Coats (Digboi); *Botany*: Prof. P. Maheswari (Calcutta); *Zoology and Entomology*: Dr. B. C. Basu (Izatnagar); *Anthropology and Archaeology*: Dr. Christoph Von Furer Haimendorf (Hyderabad); *Medical and Veterinary Sciences*: Dr. M. V. Radhakrishna Rao (Bombay); *Agriculture*: Rai Bahadur R. L. Sethi (New Delhi); *Physiology*: Dr. Kalidas Mitra (New Delhi); *Psychology and Educational Science*: Prof. Kali Prasad (Lucknow); *Engineering and Metallurgy*: Mr. D. R. Malhotra (Ajmer).

*The Association of Scientific Workers of India*: The following office-bearers for the year 1949 have been elected: *President*: Pandit Jawaharlal Nehru; *Vice-President*: Dr. B. C. Guha; *General Secretaries*: Dr. P. K. Kichlu and Mr. C. R. Mitra.

*The 5th International Congress for Comparative Pathology* will be held at Istanbul, May 17-20, 1949, and will be under the

chairmanship of Akil Moukhtar Ozden. Papers on problems of medical, veterinary, or plant pathology sciences may be submitted to: Dr. Louis Grollet, Secrétaire Général, Comité International Permanent des Congrès de Pathologie Comparée, 7 rue Gustave Nadaud, Paris, 16e, France.

*The National Registry of Rare Chemicals*, 35 West 33rd Street, Chicago 16, Illinois, is currently interested in obtaining the following "wanted" chemicals: lecithin phosphatase, *p*-tolylphosphorus dichloride, pyrosulphuryl chloride, 2-fluoroethanol, copper hydride, pelargonin chloride, *l*-borneol, mesobilirubinogen, clupanodonic acid, altrose, tagatose, *iso*-fenchyl alcohol, myrtenol, sabinol, verbenol, thujyl alcohol, bufotenidin, 5-(hydroxymethyl)-2-furaldehyde, hyodesoxycholic acid, phosphopyruvic acid, and coproporphyrin.

*UNESCO New Director-General*—M. Jaime Torres Bodet, Mexican Foreign Minister, has been elected Director-General in succession to Dr. Julian Huxley at the third General Conference of *UNESCO* held last December at Beirut.

*Nobel Prize for Medicine*—The Nobel Prize for Medicine for the year 1948 has been awarded to Dr. Paul Moller of the *Geigy Company* of Basle for his discovery of the insecticidal effect of DDT.

*Dr. James W. McBain*, Professor of Chemistry at the Stanford University, U.S.A., has been appointed Director, *National Chemical Laboratory*, Poona.

*Prof. S. N. Bose* of the Calcutta University has been elected President of the *National Institute of Sciences of India* for the year 1949.

# Reports from States & Provinces

## MYSORE

### Mineral Investigations

*The Records of the Mysore Geological Department* for the year 1945 (Supdt., Govt. Press, Bangalore, pp. iv+104, 1948, Vol. 44, price Rs. 2), includes an interesting and exhaustive paper on "Gold Investigations in Mysore" by Mr. B. Rama Rao. The paper reviews the position of India in the world with regard to gold production, and shows that out of about 1 per cent of India's share, 99 per cent comes from Mysore. It is stated that ancient miners had touched all the possible gold-bearing quartz reefs, and these, though not always rich, have provided guidance for further investigation and exploration. The Kolar Gold-fields have been developed on ancient gold-workings. In recent years there have been many explorations for gold and the laterite-covered schistose ground near Manighatta, where extensive geophysical and trenching surveys were conducted, revealed no workable gold-bearing quartz reefs. Other explorations include alluvial gold extractions in the Tungabhadra river near Holalur and Govinkere villages, and at Kudurekonda and Palavanahalli; but these also did not prove to be paying on account of water scarcity and poor concentration of gold in the washings. The explorations at Kudurekonda-Palavanahalli and Bellara, where the mode of occurrence of gold in the quartz reefs is almost similar to that at Kolar, did not turn out to be hopeful, due mainly to difficulties encountered in mining operations in areas with heavy inrush of water or the poor quality of the ore. Only sporadic occurrences of comparatively rich gold shoots and splashes occur. The paper ends with a list of localities for future investigation with geological aspects and mining possibilities. The author strikes an optimistic note about the investigations to be carried out in spite of the cost involved.

The paper "On the occurrence of road metals and lesser minerals

in Kunigal taluk, Tumkur District" is of special interest. Besides road metals, the author mentions the availability of *kankar* for extraction of lime, alluvial clays in the valleys and tanks useful for roofing and flooring tiles, and of semi-precious ruby corundum and pink garnet crystals in the gneiss and pegmatites. Amphibole asbestos is also found in small stringers in the amphibolites.

The results of geophysical prospecting for graphite near Mavinahalli, Mysore District, carried out by Mr. M. B. Ramachandra Rao, are included in the *Records*. The occurrence of a graphitic lode in a small band of ultra-basic rock amidst the thick scrub jungle had been reported previously, and geophysical technique was employed to discover the extent of the graphite lode beneath the cover of thick jungle and soil. An area of about 60 acres was surveyed by the spontaneous polarization and the earth resistivity methods along 15 lines totalling 15,400' involving 1,472 determinations. The electrical reactions obtained on the known lode were too poor and indistinct to offer any clue about the probable extent of the lode since graphite occurs therein mostly in an unevenly disseminated flaky form, which does not react as a continuous conductor. The conductive anomalies noticed in the area were due to the presence of the clayey materials with entrapped moisture which serves as a continuous ionic path for the current. The results, though not encouraging, contain valuable information about the practical details of geophysical techniques and their interpretation.

The occurrence of the felsite series near Marconahalli with a brief petrographic description of a few of the types found therein are reported in another paper. The chief types are vitrophyric, felsophyric, granophyric, and some modified types, among which there is a felspathic rock resembling the contact metamorphic rock "Unakite". It is

concluded that the felsite series are products of crystallization from an acidic magma, which later emplaced older hornblende schists and have given rise to the modified members by partial assimilation and metamorphism following granitization.

A report containing the occurrences, quality and extent of quartz deposits within an area of 15 miles' radius around Mysore, suitable for the manufacture of ferrosilicon in the *Ferro-alloy Factory*, Mysore, forms the next paper in the *Record*.

The last paper in the *Records* contains a note on "Bowenite and Talc-Picrolite intergrowth in the Ultra-basic rock from Hole-narsipur area". The optical properties and chemical analyses of talc and picrolite are given, and the latter is remarkable in having no ferric oxide but in containing about 5 per cent of aluminium oxide. The apple-green serpentine, which has greater hardness than the usual varieties, is identified to be "Bowenite".

N. V. R. SUBRAHMANYAM

### Plastics Factory for Bangalore

MACHINERY AND OTHER EQUIPMENT worth Rs. 8 lakhs for the Plastics Factory is ready for shipment in Britain. The machinery were especially designed in one of Britain's most up-to-date factories at Upper Basildon, Berkshire. The plant consists of four 3", six 2" and eight 1½" extruders, with wire covering and cable equipment, conveyor and take-off mechanisms, extruder dies and nozzles.

Two apprentices from Mysore have been trained at the Berkshire factory and three British experts will be in charge of erection of plant at the site.

## HIMACHAL PRADESH

### Fruit Development

A PROPOSAL TO BRING UNDER fruit cultivation about 7,000 acres of unused forest land in the Himachal Pradesh is under the consideration of the Central Ministry of Agriculture. A recent survey of two *tehsils* in the Himachal Pradesh showed that the area is suitable for growing walnuts, almonds, apricots, raisins, chilgoza and grapes.

# INDIAN PATENTS

The following is a list of a few of the Patent Applications notified as accepted in the *Gazette of India*, Part II, Section I, for December 1948. Patents from the Council of Scientific & Industrial Research are indicated by an asterisk.

## Plastics & Plasticizers

39251. HAPPE : Urea formaldehyde composition : *Plasticizing urea-formaldehyde resin with albuminous substances in the presence of free urea.*

## Inorganic Chemicals

37421. DELHI CLOTH & GENERAL MILLS CO. LTD., SHARMA, JAIN & CHIPALKATTI : Process for the production of raw material rich in titanium from residue of bauxite after separation of alumina : *Treatment with sulphuric acid to dissolve only aluminium and iron.*
37422. DELHI CLOTH & GENERAL MILLS CO. LTD., SHARMA & CHIPALKATTI : Process for the production of a solution rich in titanium from bauxite or residues of the same obtained after separation of alumina : *Bauxite treated with sulphuric acid till all constituents dissolve, then water added.*
37419. DELHI CLOTH & GENERAL MILLS CO. LTD., PARTHASARTHY & ARVAMUTHACHARI : Process for the preparation of chloro-sulphonic acid : *Mixing contact gas directing with HCl gas within the limits of 20 : 1 to 30 : 1 at a temperature 30° to 100°C.*
37420. DELHI CLOTH & GENERAL MILLS CO. LTD., KUMAR, PARTHASARTHY & ARVAMUTHACHARI : Process for the preparation of chloro-sulphonic acid : *Passing HCl gas in liquid SO<sub>3</sub> produced by condensing the vapours obtained by distilling oleum.*
38308. STANDARD OIL DEVELOPMENT CO. : Synthesis of hydrocarbons from carbon monoxide : *Contacting carbon monoxide and hydrogen with a relatively dilute catalyst, withdrawing the unreacted CO and H and contacting them with a dense fluidized mass of catalyst.*
30340. I.C.I. LTD. : Purification and compression of ethylene : *Forming solid ethylene hydrate and recovering ethylene from it.*
37860. E.I. DU PONT DE NEMOURS & Co. : Production of aromatic aldehydes : *Subjecting an aromatic hydrocarbon and carbon monoxide to the action of mixed hydrogen fluoride-boron trifluoride catalyst.*

## Analytical Chemistry

38754. AMERICAN CYANAMID CO. : Separation of sugar from molasses : *Passing molasses through an organolite charged with hydrogen ions, and then concentrating sugar from it.*
30888. THE ALUMINIUM PLANT & VESSEL CO. LTD. : Distillation of complex mixtures : *Distilling the mixture of acetone-methanol-water in presence of a paraffinoid aromatic hydrocarbon mixture.*
40242. ANGLO-IRANIAN OIL CO. LTD. : Refining of kerosene : *Subjecting to the treatment with sulphuric acid and then with liquid sulphur dioxide.*
38389. AMERICAN CYANAMID CO. : Purification of aqueous solutions of sugar : *Passing of solution through ion exchanger, adding about 0.01 to 0.1*

*per cent of phosphoric acid and keeping the pH near about 6-8.*

## Miscellaneous Chemicals

37426. DELHI CLOTH & GENERAL MILLS CO. LTD., GUPTA, JAGANNATHAN & CHIPALKATTI : Improved composition for preparing sizing material for textile fibres and process for making same : *Comprising rosin, glue, an emulsifying oil and a preservative.*

## Fuels & Lubricants

39057. KOPPERS CO. INC. : Coking expanding coals : *Expanding coal admixed with pitch, forming the oven charge, is surrounded by a stratum of non-coking solid fuel, and coking the charge while it is maintained stationary.*

## Leather & Leather Products

- \*38063. SIDDIQUI & DHAR : Compositions and process for the manufacture of patent leather : *Dissolving oil-resin along with film scraps in butyl acetate and solvent naphtha.*

## Metal & Metal Products

32435. I.C.I. LTD. : Copper alloys : *Copper alloy comprising nickel 9-30 per cent, manganese 5-30 per cent, aluminium 0.1-2 per cent and rest copper.*
32434. I.C.I. LTD. : Copper alloys : *Consisting of copper, manganese 5-30 per cent and iron 0.5-5 per cent and nickel 5-30 per cent.*
37205. ELECTRIC FURNACE PRODUCTS CO. LTD. : Iron-base alloys : *Containing 15-25 per cent chromium, 15 per cent nickel, 10 per cent to 25 per cent cobalt, 1 per cent to 3.5 per cent molybdenum, 0.5 per cent to 3 per cent of one or more of niobium, tantalum, and vanadium, 2 per cent manganese, 1 per cent silicon, 35 per cent carbon, 0.5 to 2.5 per cent nitrogen, characterized by having a tungsten content before 7.5 to 15 per cent, and iron content at least 15 per cent of the alloy.*

## Rubber & Rubber Products

38224. WINGFOOT CORP. : Method of making improved sponge rubber from latex : *Heating latex with 1 to 3 per cent of paraffin or wax followed by the addition of vulcanizers, gellers, anti-oxidants and accelerators at 160°-400°F.*

## Textile & Textile Products

39202. RAO : Preparation of solutions or dispersions of cellulose and their application in textile sizing, finishing, dyeing and printing : *Textile is sized, finished or dyed with a solution or dispersion of cellulose activated by metallic catalyst.*

## Miscellaneous

39431. NATIONAL RADIO & ENGINEERING CO. LTD. : Method of preparing electrodes for capacitors : *Silver nitrate mixed with water and glycerine and heated to effect reduction of silver nitrate, silver separated from glycerine being mixed with a lacquer, the paste being suitable for making electrodes.*

## “The Wealth of India”

**W**E welcome the publication of *The Wealth of India*, a new encyclopaedia dealing with the natural resources of India, in six volumes, the first of which has just reached us. The *Council of Scientific & Industrial Research*, which has taken the initiative in compiling and publishing a standard work of reference on the natural resources of India, must be congratulated on this commendable and great enterprise.

The pioneering efforts of George Watt to take stock of the raw material resources of India resulted in the publication, some fifty years ago, of the now well-known *Dictionary of Raw Materials of India* in six volumes. The *Commercial Products of India*,—which is a brief summary of his bigger work, appeared in 1908. Watt had collected together information and opinions from many sources for his compilation, and the opinions, often conflicting, were published without any attempt to critically appraise them. Later researches have proved or disproved the validity of many of the views brought together in the Dictionary. The scientific and vernacular names used were the best he could collect at that time, and these too have undergone changes. Library and laboratory facilities in India were limited during Watt's time and many of the materials briefly referred to in the Dictionary have formed the subjects of intensive study in later years. These facts led the Council to make a fresh attempt to compile a new critical compendium of data relating to the present and potential wealth of the country.

The importance of this work cannot be over-rated. India is passing through a period of rapid and fundamental changes. The somnolent agricultural country of yesterday is blossoming into an industrial State of great potentiality. Every day one hears of a large number of plans and projects undertaken by the Government and by individuals. Planning must be based on a correct and ac-

curate knowledge of the potentialities of the country, and a complete resume of the country's resources is the crying need of the hour.

The material resources of the country lie scattered far and wide, in the rich fields of the plains, in the minerals underground, in the creatures of the sea and in the plants and trees of the forests. The compilation comprehends all the information on these resources. Very often the administrator is baffled by conflicting and contradictory opinions given by different workers on the same topic. In this newly planned Dictionary, an attempt is made to critically assess all opinions and to present a carefully integrated view supported by scientific reasoning. Statistical information, wherever available, is also presented in carefully arranged tables.

At present, various departments of the Government are engaged, incidentally, in collecting together information relating to their specialized fields. Thus, the Forest Research Institute, in addition to its regular research work, collects all information on forest products. The Indian Council of Agricultural Research, through its several schemes and Commodity Committees like the Cotton Committee and the Sugarcane Committee, is doing similar work on crops and plants. The Central Agricultural Marketing Department publishes frequently various reports dealing with surveys and standards for trade of particular commodities. The Tariff Board carries out detailed inquiries into industries and makes suggestions to the Government on price structures, duties and tariffs. The Zoological Survey and the Geological Survey conduct researches in their particular fields and publish memoirs on the results achieved. There are a number of research journals published by learned societies and research institutions located in various regions of the country. Any individual interested in a particular problem has, at present, to go through a maze of papers

and sift for himself the information which he requires. Thus, a scientist working on an economic plant may have to consult the Forest Research Institute publications, Tariff Board reports and several scientific papers for acquainting himself with the work done on the different aspects of the plant. The assembling of information in the encyclopaedia would provide a valuable aid to him in his search, while for details relating to techniques and results, he would have to still look to original papers as source materials. For the scientific worker, this book will also be a good guide to bibliography.

The office of the Dictionary of Economic Products is fast becoming a store-house of information on divergent subjects. For each topic, a separate ledger containing all available information is opened. A batch of workers, combing the journals and periodicals published here and abroad, is carefully adding

to this pool of knowledge. The ledgers are thus kept "alive" and can be consulted by anyone interested. We have in these columns repeatedly stressed the necessity for organizing a Central Scientific Information Service for India. There is every reason to hope that the Council's organization for compiling the Dictionary would form the necessary nucleus for building up such a Service.

The work is of immense value to the industrialist. He will get at one glance a correct perspective of the present state of the industry he is interested in — its drawbacks and its potentialities. He can base his further plans of improvement on this knowledge.

The task undertaken by the Council has great potentialities. In the present stage of the country's development such an encyclopaedia is an absolute necessity, and it is hoped that the Council would complete the work expeditiously.

## Scientific Information

THE recommendations relating to the preparation of scientific communications adopted at the last meeting of the Royal Society Scientific Information Conference (June-July 1948) are of wide interest. The Conference called upon all those handling papers in draft form, including heads of departments in universities and technical colleges, and directors of Government laboratories, research associations, and industrial laboratories to do their utmost to ensure that :

"Manuscript submitted by authors under their direction is written in clear and concise English, and is prepared in accordance with the rules of the journal for which it is intended.

To the best of their knowledge and belief, any contribution passed through their hands, whether as communicator or referee, is worthy of publication on its merits as scientific information alone.

They would help their students and assistants in preparing such material.

Special efforts are made to save time at all stages of preparation, from the completion of actual research work to final printing, so as to avoid the psychological effect of dilatoriness on all those involved in the chain of publication.

Scientists, as potential authors, should be trained in the initial preparation of manuscripts designed for publication, so as to reduce subsequent corrections by printer and author to a minimum, and thus save the time of scientific editors. The need for layout and expression should be emphasized. For reproduction by photo-lithography this is of particular importance."

The Conference recommended that critical and constructive reviews, written in particular fields, and reviews by specialists for other workers in science, are of great importance.

Critical, general and specialist reviews should be made informative to non-specialists by a general introduction and conclusion.

Senior scientists should regard the provision of reviews as an important ancillary to the pursuit of new knowledge.

Annual reviews, progress reports and books on recent advances covering limited fields in a comprehensive but critical manner are very valuable and serve a purpose different from that of reviews. Their production should be encouraged.

The attention of the appropriate societies, institutions, and Government organizations should be drawn to the importance of providing reviews on the progress of applied sciences.



# The Patent System & the Scientist\*

K. RAMA PAI

A "PATENT" is in the nature of a contract between the State and the inventor. Under the contract, the inventor discloses to the State what he believes to be a new invention; and in consideration of the disclosure made by the inventor, the State grants him a "patent" for the invention disclosed. The effect of granting a patent is to confer on the grantee certain exclusive rights in respect of the patented invention for a limited period, which is usually 16 years.

It is understood that the information disclosed by the inventor would be published by the State even before the patent is granted, and that on the expiry of the patent, the invention would become dedicated to the public for free use by any one without being under any obligation to the patentee.

The result of such disclosure is to render the patent office a central treasury of inventions and technical information. A question may, therefore, be asked in this connection whether the scientist also has not a duty to bring his inventions on the records of the patent office, and thereby add to the wealth of information available in the nation's treasury of inventions. As thousands of inventions are disclosed every year to the various patent offices, patent literature constitutes the most exhaustive form of technical literature pertaining to any and every branch of industry. I believe, this is a matter which has not been adequately appreciated in this country; for, so far as I am aware, there is no research centre in India where the patent literature of industrially advanced countries such as the United States of America, the United Kingdom, or Germany can be had for convenient reference.

Another matter which deserves notice is that industrialists, when they are on the look out for inventions, invariably consult the patent literature of the industry concerned. Accordingly, publication of inventions through the Patent Office brings such inventions to the notice of the very people

who are likely to be interested in them, and, therefore, ensures for such inventions the most effective form of publicity.

Next, I would refer to the "exclusive right" conferred by a patent. This right is given only in exchange for two privileges surrendered by the inventor, namely:

- (i) the prompt disclosure of the invention to the public; and
- (ii) the unreserved dedication of the invention to the public on the expiry of the patent.

What the patent system actually does is merely to restrict the period of exclusive right to a reasonable period of 16 years. The patent does not compel the patentee to exercise his exclusive right. An inventor can allow the public to use his invention freely even after patenting it, or use his patent rights to regulate the manufacture and the sale of the patented article in the best interests of the public, as was done by the Toronto University in connection with their patent for insulin.

A new invention, as soon as it is evolved, is not readily adaptable for industrial purposes. It requires to be developed at considerable expense and labour. If, when this development is made, the person who takes the trouble to develop the invention cannot have, even for a short period, some privileged rights over the invention as compared with others who had been indifferent to the invention, there would be no incentive for anyone to develop new inventions.

I would now consider why scientists should concern themselves with the patent system. In this connection it is necessary to be clear as to the rôle of science in the present day. The traditional rôle of science has been the search of knowledge for its own sake. Nevertheless, society looks up to the scientist not only to expand the frontiers of knowledge, but also to solve numerous problems which face it, such as the economic problem of finding food and employment, the defence problem of maintaining an adequate war potential which would ensure freedom to the

\* From contribution to the Symposium on the Patent System held during the Annual Session of the Indian Science Congress, 1949.



nation, and a thousand and one other problems which would assist men in passing through life with maximum comfort in return for the minimum of effort. Accordingly, scientific research includes not only fundamental research which discloses to us nature's secrets, but also applied research which enables us to make use of the discoveries resulting from fundamental research.

Hence, the best results out of scientific research would accrue to society only if the maximum advantage is taken of discoveries of both categories of research. If this is accepted, it would also follow as a natural corollary, that the scientist has a *duty* to concern himself with every factor which would be helpful to him for adapting his discoveries in the field of applied research, for utilitarian purposes.

The patent system has been recognized as one of the most powerful factors which would encourage inventors to develop inventions from the laboratory stage to the industrial stage; and, therefore, in order that discoveries of utilitarian value may be used for the maximum advantage of the society, it is incumbent on the scientist to take full advantage of the patent system for developing his inventions for industrial purposes.

I would now refer to a few instances which would show how the country cannot get the best out of her research resources so long as the scientist is indifferent to the patent system. In the first place, I would refer to the neglect of patent literature. As a result of this neglect, it has been noticed that in numerous instances scientific research workers have been merely re-inventing what had been already invented by others. To the extent that the resources of research have been utilized for re-inventing what has already been invented by others, the inventor has been wasting the industrial resources of the country. He can avoid such waste merely by consulting the patent literature before he embarks on his research.

Next, I would refer to an instance where an Indian scientist invented a very useful machine. He sent the description of the machine to a scientific journal for publication. As soon as the description was published, one of the British instrument manufacturers got interested in the invention, and asked for the inventor's terms for obtain-

ing from the inventor an assignment of his patent rights for the invention. But the inventor had not taken out any patent; nor could he apply for a patent after the publication of the invention. Accordingly, as the result of premature publication of the invention, a commercial transaction which would have brought money not only to the inventor, but also indirectly to the country, could not materialize. The question may, therefore, be asked whether scientists are justified in thus causing an economic loss to the country by their indifference to the patent system.

Next, there is the question of the development of the invention. It was found in other countries that inventions of utilitarian value were developed under patent protection and that without patent protection they were not adopted for industrial purposes even if they had immense potentialities. At present, no systematic survey has been made by anyone to see to what extent results of applied research in India have been developed beyond the laboratory stage. But, to the extent that I have had personal experience, I have found that in the case of patented inventions, serious attempts are invariably made for developing the inventions for industrial purposes; but where the inventions have not been protected by a patent, they are invariably abandoned at the laboratory stage. I consider, therefore, that the neglect of the patent system by the Indian scientist results at present in a huge waste of the country's industrial and economic resources.

India has embarked upon a scheme of research on an unprecedented scale, and there can be no doubt that many inventions of a high order would be made by the Indian scientists. If such inventions were merely broadcast for free use by anyone who would like to use them, it would be like scattering good seedlings on the highway in the hope that somebody would pick them up and take the trouble of planting them on proper soil and fostering them until they grow up into budding plants.

In order to ensure that the country gets the best advantage out of our slender resources for industrial research, it is urgent, therefore, that a carefully chalked out policy should be formulated for the guidance of scientists engaged in industrial research.

## The Board of Scientific & Industrial Research Twenty-second Meeting, New Delhi

A MEETING of the Board of Scientific & Industrial Research was held in New Delhi on 18th February 1949. The Hon'ble Pandit Jawaharlal Nehru presided.

On the recommendation of the Board of Scientific & Industrial Research, the Governing Body approved the continuance of over hundred schemes of research investigations covering a wide range of subjects. The following new schemes were recommended for sanction to the Governing Body :

1. Preparation of drugs containing coumarin and chromone nucleus : DR. D. CHAKRAVARTY (*Calcutta*).

2. Construction of an osmometer for high polymer research: DR. S. R. PALIT (*Calcutta*).

3. Utilization of cellulose resources other than cotton : MR. D. L. SEN (*Bombay*).

4. Insecticides from tobacco : DR. T. R. SESHADRI (*Waltair*).

5. Chemistry and pharmacy of Indian ergot : DR. T. R. SESHADRI (*Waltair*).

6. Survey of fungus flora as sources of antibiotics : DR. S. R. BOSE (*Calcutta*).

*Central Food Technological Research Institute* — The Prime Minister announced the gift of the Cheluvamba Mansion in Mysore for housing the Institute. Necessary funds have now been sanctioned by the Government of India to equip the buildings with laboratory facilities and services. Research work on technological aspects of the country's food problems will soon be taken up at the Institute for investigation.

*Central Drug Research Institute* — The Government of the United Provinces have offered the historic mansion "Chattar Manzil" at Lucknow for housing the Institute. The building has a floor area of 60,000 sq. ft. and about 80 rooms in its 3 storeys.

With the establishment of these two Institutes, the first phase in the founding of a chain of 11 national laboratories in the country initiated by Dr. S. S. Bhatnagar will be nearly complete.

*Dictionary of Raw Materials & Industrial Resources of India* — The publication of the first volume of the dictionary, *The Wealth of India*, was announced. The work will comprise of six volumes and deal with 4,000 subjects covering the natural resources of

India. The first volume covers the letters A and B and runs to 400 pages.

*Low-temperature Carbonization of Coal* — Among the proposals adopted by the Governing Body of the Council was the scheme to set up plant-scale investigations for low temperature carbonization of coal at the *Fuel Research Institute*, Dhanbad. By the low-temperature carbonization of 3.5 million tons of coal, which amount has been estimated as necessary for meeting India's soft coke requirements, enough by-product tar and oils would be available for the production of nearly 200,000 tons of synthetic petroleum and the by-product gas produced could be utilized for domestic and industrial heating purposes. The proposed plant at Dhanbad (capital cost Rs. 10 lakhs) would carry out investigational work for adoption of this process to Indian conditions.

*Optical Glass Manufacture* — The Board accepted the recommendation of the Glass & Refractories Research Committee on the production of optical glass in India. It has been recommended that investigational and pilot-plant work should be taken up without delay at the Council's Central Glass & Ceramic Research Institute, Calcutta. It has also been recommended that a team of ceramic and glass experts should visit the various ceramic and glass industrial centres in the country and make a survey of their difficulties and problems and report to the Government of India as to what steps should be taken to improve the conditions in the industry and what help should be given for its scientific development.

*Radio Research* — It has been proposed to set up under the auspices of the Council an ionospheric field station outside the city of Calcutta at a cost of about Rs. 50,000. The station will carry out research work on ionospheric conditions over Calcutta and will work under the supervision of the Radio Research Committee of the Council.

*Malaria Chemotherapy Committee* — The Governing Body has approved of the proposal to constitute a standing Malaria Chemotherapy Committee under the Pharmaceutical and Drugs Committee which would co-ordinate and direct research on malaria prevention and cure now carried out in the country.

## Exhibition of Scientific Instruments

THE exhibition of scientific instruments, which the Indian Science Congress organizes at its annual sessions, provides an opportunity to assess the progress achieved by the scientific instruments industry in India. A large number of well-established firms are now manufacturing many of the instruments and apparatus which were being imported during the pre-war years.

As many as 15 firms took part in the exhibition, and a wide range of instruments and apparatus used in teaching and research in physical, chemical, and biological sciences were on show. In design and quality of workmanship also there has been a remarkable advance. The exhibits included: cathetometers, spectrometers and spectroscopes, optical benches, reading and research microscopes, telescopes, galvanometers, dip-needles, and other electrical measuring instruments, several new types of balances, optical lenses, laboratory glassware and others. The *Andhra Scientific Co. Ltd.*, Masulipatam, had exhibited instruments which attracted much attention. A wave-length spectrometer with camera attachment and wave-length drum, designed and fabricated in their own workshops, was shown. This firm manufactures arc lamps and other accessories for advanced spectroscopic work. Their Admiralty Pattern Sextant, manufactured for the first time in India, was a notable addition to the range of instruments. A specially designed aberrationmeter for testing inaccuracies and defects in lenses was also exhibited.

A wide range of instruments specially designed to meet the requirements of educational institutions was exhibited by *Messrs Scientific Instruments Co.*, Allahabad; *Instruments & Chemicals Ltd.*, Ambala; and *Scientific Apparatus Manufacturing Co. Ltd.*, Agra. These exhibits were characterized by sturdy construction and simplicity of design. Dip-needles, galvanometers, potentiometers, verniers, induction coils, resistance boxes, Wheatstone bridges, optical benches, etc., were some of those under this head. The Agra firm specializes in instruments such as kymograph apparatus, myographs, time markers, recording drums, respiration pumps, etc., required in pharmacological and physiological research.

Lenses, optical instruments and accessories are now being manufactured by the *Instruments Research Laboratory*, Calcutta. Optical glass is imported in bulk quantity from abroad and cutting, shaping, grinding and polishing are carried out in the firm's workshop. There is a considerable demand in India for achromatic lenses, low-power microscope objectives, prisms of various types, optically flat glass surfaces, magnifying lenses, etc., and they are now being satisfactorily met by the *Instruments Research Laboratory*.

Two other exhibits which attracted attention were a miniature gas plant manufactured by *Messrs Gansons Ltd.*, Bombay, and a new type of chemical balance with an arrangement for the mechanical manipulation of fractional and gramme weights (sensitivity  $\frac{1}{10}$  mg.) manufactured by the *Balance Works*, Banaras. The gas plant is a compact, self-contained unit working on petrol with an automatic control mechanism for gas supply. The manufacturers can supply plants of various capacities, the largest being able to feed 500 burners.

Mention should be made of *Messrs Kaycee Industries Ltd.*, Poona, who had exhibited an extensive range of electrical measuring instruments of good quality, design, workmanship and finish.

The instruments exhibited during the recent session of the Indian Science Congress were those marketed by instrument manufacturers in the country. In a highly specialized field of this type, there are a large number of instruments developed by research workers primarily to meet their special requirements, but which have potentialities for wider use. A large number of instruments based on research and constructed by skilled craftsmen are in use in specialist organizations, e.g. Indian Meteorological Department, Mathematical Instruments Office and Civil Aviation Directorate. For a proper appraisal of the progress in the design and fabrication of scientific instruments, all organizations interested in scientific instruments should be invited to participate in the exhibition. We hope that in the coming session the delegates would have the opportunity to see a wider range of exhibits and to discuss with designers and manufacturers the special problems relating to their requirements.

# Characteristics of the Ionosphere over Calcutta (December 1948)

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THE following are the ionospheric data observed at Calcutta for the month of December 1948. The observations were made at each hour of the day for 5 days a week.

Fig. 1 presents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form during the month of December 1948. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmission during March 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when the E region was found to be abnormal and the corresponding penetration frequencies and heights.

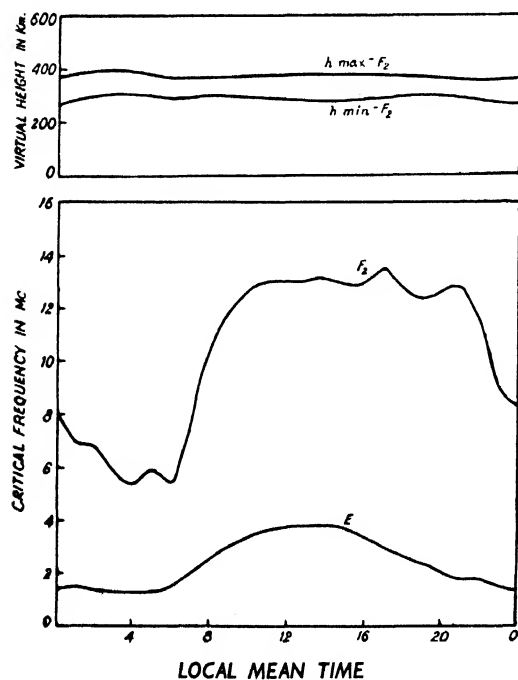
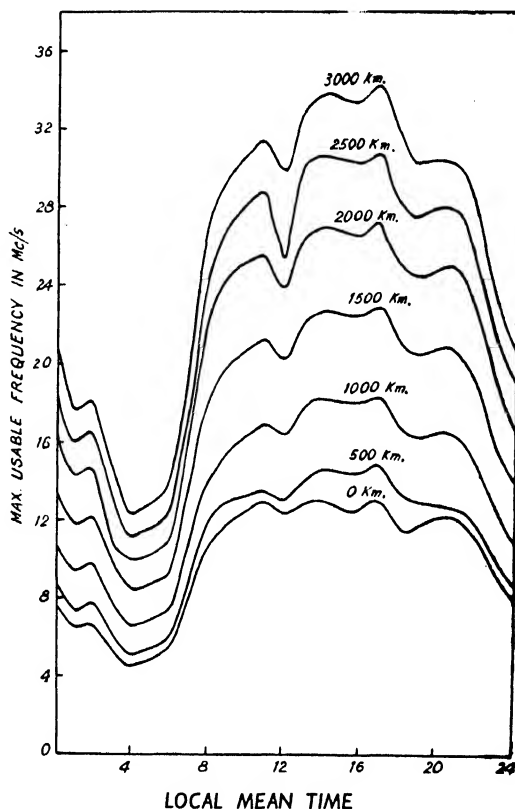


FIG. 1 — DECEMBER 1948. 5HR. 54 MIN. AHEAD OF G.M.T.



AT POINT OF REFLECTION.  
FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION VIA  $F_2$  LAYER, MARCH 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^o E_s$	$h E_s$
			Mc.	Km.
Dec. 1948	1	17.00	3.30	105
		18.00	3.00	105
		19.00	2.55	90
	3	18.00	2.65	105
		19.00	2.55	105
		22.00	2.25	90
		23.00	2.25	90

TABLE I—contd.

MONTH & YEAR	DATE	HOURL	$f^\circ E_s$ Mc.	$\lambda E_s$ Km.
Dec. 1948	7	00.00	2.30	90
		01.00	2.55	90
		02.00	2.70	90
		03.00	2.80	90
		04.00	2.95	90
		05.00	2.50	105
		07.00	2.25	105
		08.00	3.30	105
		09.00	3.35	105
		10.00	3.50	105
		16.00	3.30	120
	9	17.00	7.00	135
		18.00	6.00	135
		19.00	5.20	120
		20.00	2.30	105
		21.00	2.25	105
		22.00	2.30	105
	10	03.00	2.50	90
		04.00	2.50	90
		05.00	3.15	105
		06.00	3.00	105
		07.00	3.15	105
		09.00	3.50	105
		16.00	3.50	105
		17.00	3.40	105
		18.00	3.15	105
		19.00	3.00	105
	13	21.00	2.30	90
		22.00	2.15	90
		23.00	2.00	90
		16.00	4.45	120
		17.00	3.20	120
		18.00	3.00	105
		19.00	3.00	105
		20.00	2.60	90
		21.00	2.00	90
		22.00	3.25	90
		23.00	2.85	90
	14	01.00	3.00	90
		02.00	4.00	105
		03.00	3.30	105
		04.00	3.25	105
		05.00	2.85	90
		06.00	2.80	90
		07.00	3.80	105
		18.00	3.00	105
		20.00	2.45	90
		21.00	2.05	90
	15	17.00	2.90	105
		18.00	2.50	105
	16	14.00	4.50	120
		17.00	4.50	120
		18.00	2.85	120
		20.00	2.50	105
		21.00	3.00	105
		22.00	2.70	105
	17	01.00	2.50	90
		05.00	2.40	90
		06.00	2.25	90
		07.00	3.00	105
		08.00	3.00	105
		09.00	3.50	105
		10.00	4.10	120
		18.00	3.35	120
	20	20.00	2.60	90
		21.00	2.65	90
		22.00	2.20	90
		23.00	2.30	90

TABLE I—contd.

MONTH & YEAR	DATE	HOURL	$f^\circ E_s$ Mc.	$\lambda E_s$ Km.
Dec. 1948	21	02.00	2.00	90
		05.00	2.30	90
		07.00	2.60	90
		08.00	3.45	105
		10.00	3.75	105
		11.00	4.10	120
		12.00	4.40	120
		13.00	4.25	120
		14.00	4.10	120
		16.00	3.55	120
	22	17.00	3.00	105
		18.00	2.50	105
		20.00	2.20	105
		22.00	2.10	90
		23.00	2.35	90
		00.00	2.30	90
	23	03.00	2.00	90
		14.00	4.35	120
		15.00	4.70	120
		16.00	4.45	120
		17.00	4.25	120
		11.00	4.00	135
	24	13.00	4.40	135
		14.00	4.00	120
		22.00	3.20	90
		23.00	4.50	90
		00.00	4.00	90
		02.00	4.00	90
	28	04.00	2.80	90
		05.00	2.50	105
		06.00	2.40	105
		07.00	2.85	105
		08.00	4.00	90
		09.00	4.00	90
		04.00	2.80	90
		05.00	2.50	105
		06.00	2.40	105
		07.00	2.85	105
	29	08.00	3.80	105
		17.00	3.85	120
		18.00	3.20	120
		19.00	2.75	105
		21.00	2.60	105
		22.00	2.25	90
	30	22.00	2.90	90
		23.00	3.05	90
		00.00	2.25	90
		01.00	2.00	90
	31	02.00	2.00	90
		10.00	4.25	105
		11.00	4.50	120
		12.00	4.25	135
		13.00	4.50	135
		14.00	4.35	135
	31	15.00	4.25	120
		16.00	4.00	120
		20.00	4.80	120
		21.00	4.00	105
		22.00	2.40	105
		23.00	2.00	90
		00.00	2.00	90
		03.00	4.70	105
	31	04.00	3.15	105
		05.00	2.40	105
		06.00	2.65	105
		07.00	2.70	105
		08.00	4.20	120
		09.00	4.20	120

# Characteristics & Composition of Vegetable Fats in Relation to the Habit of the Plant

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**P**LANTS, bearing seeds which yield important vegetable fats, belong to various families and exist in different statures. From an examination of the characteristics and compositions of these fats, several important conclusions have been drawn. For instance, seeds from plants of the same or of closely related natural orders furnish oils containing the same fatty acid. Certain families produce seeds yielding fats predominating in a particular fatty acid or acids, e.g. myristic acid in Myristicaceae, lauric acid in Lauraceae and elaeostearic acid in Euphorbiaceae, etc. It has been recognized that the degree of unsaturation of the fat is dependent upon climatic conditions<sup>1</sup>, especially temperature, to which the plant is exposed. The sensitiveness of the plant to variations in climatic conditions is particularly conspicuous in plants, the seeds of which contain unsaturated fatty acids with 2 or 3 double bonds.

No definite attempt has so far been made to correlate the characteristics and the composition of seed fats with the stature of the plant. In the course of our work<sup>2</sup> on the fatty acid composition and glyceride struc-

ture of vegetable fats from different botanical families, a striking relationship was observed between the habit of the plant and the degree of unsaturation of the fatty acid constituents of the seed fats. Tree seeds, generally, yielded fats of a lesser degree of unsaturation, while seeds from herbs and shrubs gave oils of an unsaturated nature. It was considered of interest to examine systematically the available data on the characteristics and composition of oils with special reference to the degree of unsaturation. The data for seed oils of the following families have been analysed: (1) Dipterocarpaceae, (2) Myristicaceae, (3) Guttiferae, (4) Lauraceae, (5) Sapotaceae, (6) Anacardiaceae, (7) Sapindaceae, (8) Meliaceae, (9) Myrtaceae, (10) Apocynaceae, (11) Linaceae, (12) Solanaceae, (13) Papaveraceae, (14) Pedaliaceae, (15) Compositae, (16) Acanthaceae, (17) Tiliaceae, (18) Malvaceae and (19) Papilionaceae.

The first ten families consist of trees, while the next seven consist of herbs or shrubs only. The last two, namely Malvaceae and Papilionaceae are interesting in that they consist of members of all statures. It can be seen from the data presented in Table I

TABLE

S. No.	NATURAL ORDER	NUMBER OF PLANTS STUDIED (REFERENCE TO LITERATURE IN BRACKETS)	HABIT	VARIATION IODINE VALUES
1	Dipterocarpaceae	Four (2-5)	Large trees	32.3-42.80
2	Myristicaceae	Thirteen (3, 6-10)	Trees	1.72-61.0
3	Guttiferae	Twelve (3, 11-17)	Trees and small trees	30.7-93.5
4	Lauraceae	Eleven (3, 6, 8, 18-21)	Trees and small trees	4.0-86.4
5	Sapotaceae	Eleven (3, 12, 22-27)	Trees	32.9-60.7
6	Anacardiaceae	Four (55-58)	Trees	39.2-97.8
7	Sapindaceae	Three (59-61)	Trees	43.8-58.5
8	Meliaceae	Two (62, 63)	Trees	72.6-74.3
9	Myrtaceae	One (64)	Tree	96.4
10	Apocynaceae	Three (65-67)	Trees	68.2-87.6
		One (65)	Trees or shrubs	145.4
11	Linaceae	Seven (28-31)	Herbs (annual)	182.3-196.9
12	Solanaceae	Twelve (2, 32-39)	Herbs or small trees	111.7-150.2
13	Papaveraceae	Two (68, 69)	Herbs	121.0-139.8
14	Pedaliaceae	Two (70, 71)	Herbs	110.8-114.0
15	Compositae	Five (72-76)	Herbs	111.3-141.3
16	Acanthaceae	One (77)	Herb	135.0
17	Tiliaceae	One (78)	Herb	109.2
18	Malvaceae	Fifteen (2, 38, 40-49)	All statures	34.7-108.9
19	Papilionaceae	Eight (8, 50-54)	All statures	63.3-123.3

TABLE II

Mean iodine value	{ Shrubs (average of 42 values)	131.94
	{ Trees (average of 74 values)	52.56
Mean difference		79.38
S.E. of mean difference		29.4
't' value for 114 d.f. of		13.9

that the seed fats derived from trees are generally more saturated than those derived from herbs or shrubs. The available data has been subjected to statistical analysis (TABLE II). The iodine values, representing the degree of unsaturation of the fat, have been tabulated according to the statures of the plant under two categories: (a) trees, and (b) shrubs and herbs. A statistical analysis<sup>4</sup> of the data shows that the mean difference in iodine value between the two groups of plants is of the order of 79.38. The analysis further indicates that the chances of a difference as large as that observed occurring through pure chance or random sampling alone would be less than 1 in 100. It may be confidently concluded from the analysis that the seed fats derived from trees are generally more saturated than those derived from herbs or shrubs in general.

TABLE III

S. No.	NATURAL ORDER	NUMBER OF PLANTS STUDIED (REFERENCE TO LITERATURE IN BRACKETS)	HABIT	VARIATION IN IODINE VALUES
1	Coniferae	Nine (80-87)	Trees	101.3-156.3
2	Euphorbiaceae	Six (88-91)	Trees and small trees	129.2-171.0
3	Juglandaceae	Ten (92-100)	Trees	97.1-161.7

It must be observed, however, that the conclusion is true only in the case of those seed oils which contain the usual types of acids. Euphorbiaceae, for example, is exceptional in possessing a few species which elaborate unusual acids. Thus ricinus species contain ricinoleic acid, and two species of aleurites yield elaeostearic acid. Further, our observation does not hold for plants originally belonging to temperate climates, e.g. pines, walnut, beech, etc., which yield drying oils though they are all large trees (Table III).

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## Silica Gel—Its Reactivation & Indication

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**S**ILICA gel is a common desiccant used for the preservation of optical and telecommunication stores in transit and storage in tropics, primarily because it is neutral in character and can be reactivated and used a number of times. It is usually supplied as a white granular solid ; sometimes a portion of the gel is coloured with cobalt chloride which acts as an indicator as to the condition of the desiccant. The granules stained with cobalt chloride appear deep blue when the gel is fully active and turn pink when saturated with moisture, thus indicating that the gel needs activation.

### Reactivation Methods

Used silica gel can be reactivated for further use by heating under controlled conditions above a temperature of 300°F. If an indicator is present, it should not be heated to temperatures above 450°F. White gel may be heated to higher temperatures without appreciable damage, but a temperature of 300° to 400°F. is usually sufficient for regeneration of all types of silica gel, the charges being normally baked for 2 to 3 hours at this temperature. On account of the poor heat conductivity of the material, it is important that during the reactivation



process it should be spread in thin layers and, if possible, frequently stirred to ensure uniform heating. Further, during the first hour of reactivation, the temperature should not be allowed to exceed 250°F. to obviate the possible breaking up of the granules.

For bulk regeneration, specially constructed electric ovens with thermostatic control are ideal. The ovens should be fitted with fine wire-mesh trays on which the gel is spread out in layers of about quarter-inch thick. The ovens should have air vents at the top and bottom to provide free circulation of air through the chamber and to facilitate escape of moisture during the reactivation process. The temperature of the oven should be regulated to 250°F. during the first hour and then increased to 400°F. and maintained at this value for a further two hours.

There may be, however, numerous occasions when the necessary equipment of the type mentioned above is not available. A *sigri*, stove or other source of heat available can be used for the purpose, the gel being roasted on a thick metal (e.g. iron) plate placed over the source of heat. The gel must be constantly raked on the tray to prevent over-heating.

#### Indication of Active Silica Gel

Two methods are usually employed to observe visually the state of exhaustion of the silica gel employed in a container and to know when it needs reactivation:

1. A portion of the gel (not enclosed) is coloured with cobalt chloride solution.

2. When the gel is enclosed in a linen bag or kept in a metal container with a perforated lid, a dab of cobalt chloride

solution on the outside of the linen bag or a thin filter paper strip dyed with the indicator solution and stuck on the perforated lid will provide the necessary indication. A 30 per cent solution of cobalt chloride for dyeing the filter paper gave the best results. A sufficiently strong and easily distinguishable tint develops at low humidity values without leaving a deposit of excess salt over the surface of the paper.

The transition of colour from blue to pink of the indicator, in both the methods employed, takes place when the relative humidity of the surrounding space reaches 60 per cent. At this stage, the silica gel still possesses an appreciable reserve of absorption capacity without unduly raising the humidity of the container to a value at which fungus growth can take place. The above change in colour of the indicator may, therefore, be considered as a safe limit when the gel has to be reconditioned.

A useful method of checking the condition of a desiccator is to determine the time taken for the indicator to change from pink to blue when the desiccator is closed. The experiments with different desiccants showed that this time should not exceed 15 minutes in the case of silica gel and quicklime desiccators and 10 minutes in the case of desiccators using quicklime-calcium chloride mixture. If the time required for the change of colour is within these limits, the desiccator may be considered to possess useful life.

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# Recent Advances in Exploration Geophysics\*

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**D**URING the past few years considerable progress has been made in America in the design of special geophysical instruments suited to exploration programmes. These developments have been achieved as a result of the co-operation of several governmental departments and private organizations whose physicists, electronic engineers, military scientists, geologists, geophysicists and photographic specialists jointly or severally contributed their technical knowledge and skill to evolve these useful designs. Some of the developments may be said to have been largely a result of the stimulus provided for research during the war. Till recently very little had been published on these developments for reasons of military security, and some secrecy still exists on the design and applications of a few of the latest instruments and techniques. However, a good deal of information has already been released to the public and a brief account of the nature of the instruments and their operation are given in this paper. The possibilities of applying these methods for geophysical exploration in India are indicated.

## Prospecting from the Air

The idea of making physical measurements from the air, high above the ground surface, is by no means quite novel. For a long time scientists have been recording some of the physical data in balloons and aeroplanes. Geophysicists were not slow in realizing the advantages of making surveys of gravitational and magnetic field variations of the earth from the air. But the problems of instrumentation suited to this type of measurement had not been solved, and as far as the gravimetric method is concerned, the problem still remains unsolved. During the war, magnetic airborne detectors were designed in U.S.A. which were successfully used for the location of enemy submarines. Further researches have resulted in the development of airborne magnetometers

which have now become well known and extensively used for geophysical surveys. By the special permission granted by the Director of the *U.S. Geological Survey*, and also the Director of Mines and Resources in Canada, the author had an opportunity of studying this new method of magnetic prospecting from the air in actual flights with the geophysical parties.

The airborne magnetometer used by the *U.S. and Canadian Geological Surveys* consists of a "flux-gate" mechanism. It is capable of automatic orientation in the magnetic meridian so that changes in the movement, mechanical oscillations, etc., of the plane are without any effect. The detector element is housed in a streamlined bomb-shaped case which is called the "bird". The bird is towed beneath the aeroplane by means of a non-magnetic cable 100' long, and is released or wound by means of a winch within the plane. The signals picked up by the magnetic element in the bird are carried to the recording meter by the towing cable itself. The total magnetic intensity of the ground in the path of the plane is measured and continuously registered electronically on a panel which is suitably constructed within the plane. The sensitivity of the magnetometer can be varied by selector switch controls.

The position of the plane in space is determined by obtaining a terrain photograph vertically above the path of flight by means of a gyroscopically stabilized continuous strip camera. A recording radio-altimeter also continuously measures and registers the height of the plane above ground. All the 3 records, namely the total magnetic intensity graph, the terrain photograph and the altimeter chart are co-ordinated by an electrical system. By matching the ground strip with the photographs on maps of suitable scale, and also knowing the height of the plane above the ground surface, it is possible to locate the traverse lines and to plot the total magnetic intensity variations along these lines. A special

\* From the lectures delivered at the *Geological Survey of India*, Calcutta, July 13-14, 1948.

mechanical device is usually employed for directly transforming the field curves of magnetic intensity into orthogonal co-ordinates.

When the flights are made over water, ice or unmapped territories, the locations are obtained by the use of "Shoran", a radio-navigation aid, or radar triangulation. With this system the distance from two ground stations is measured electronically and is constantly recorded and co-ordinated with the total magnetic intensity chart. The traverse lines could then be deduced, knowing the position of the ground stations, and the magnetic intensity variation plotted with reference to such lines.

There are some modifications in the construction of the magnetic elements, the recording devices and other details in the airborne magnetometer units which have been developed by some of the private companies operating for regular surveys on a commercial basis. For instance, the system built by *Heiland Research Corporation* and used by the *Geophysical Exploration Company* of Denver, Colorado, has a detector unit of small dimension which is built in the wing tip of the plane itself. Dr. Hans Lundberg's organization has a separate type of magnetometer which is mounted in front of the cabin in a helicopter. The heli-

copter type of aircraft has great manoeuvrability as it could fly at very low altitudes. Its speed is less, and a given point could be reached at a given moment, and it is also possible to keep hovering at a point, ascend or descend vertically over a fixed point. It is capable of landing in small clearings.

The advantages of the airborne magnetometric surveys are the obvious speed with which the measurements are made and the ability to gain knowledge of the remote inaccessible areas, especially over swamps, lakes, deserts, jungles, and mountainous tracts. The airborne measurements also effectively eliminate — or at least minimize, the interferences caused by rails, pipes, transmission lines and other disturbing factors met with in the ground surveys. To the advantages offered by the airborne unit, we may also add the ability to study a particular indication or anomaly at different altitudes and thus obtain valuable additional data for interpretation.

The U.S. *Geological Survey* crews have carried out, during 1946, airborne magnetometer surveys over an aggregate area of over 50,000 sq. miles including portions of the oil-fields of Northern Alaska. A good part of their more recent surveys have been done over the metamorphic and

igneous rocks in the iron country, Michigan, and in the Adirondacks. The purpose of the surveys was the location of iron ore deposits, and the results are reported to be successful. They have also carried out airborne surveys for locating sedimentary structures in the Big Horn Basin, Wyoming; the Atlantic coast plain, Maryland; and also some places in Oklahoma State; but the results of these surveys were said to be of rather indifferent success. Several aeromagnetic surveys by private companies are reported to have been carried out in the search for sub-surface ridges of igneous rocks which are associated with certain types of oil-field; the results of these surveys are not known.

In Canada, the *National Research Council*, the *Geological Survey* and the *Royal Canadian*



FIG. 1 — A BEECH-CRAFT AEROPLANE TOWING THE BOMB-LIKE "BIRD" IN WHICH A SENSITIVE MAGNETOMETER IS KEPT. THE IMPULSES DUE TO THE EARTH'S MAGNETIC FIELD ARE PICKED UP BY THE TOWING CABLE TO A RECORDING INSTRUMENT INSIDE THE PLANE.

(By courtesy of "Engineering & Mining Journal", McGraw-Hill Publication, New York.)

*Air Force* have co-operated to carry out airborne magnetometer surveys in parts of the Canadian Shield. About 1,200 sq. miles of the Carleton Place, Arnprior and Perth area in Ontario Province have been covered and some indications over the iron ore bodies have been obtained. Small areas have also been surveyed in the Sudbury areas (Ontario) and the Louvicourt township in Quebec.

Although some notable successes have already been achieved in the practical application of magnetic prospecting from the air, and increasing attention is being paid to the question of extending such surveys over large territories, the serious limitations in the use of this method are also being clearly recognized. The airborne device cannot dispense with the ground magnetometer measurements which should be carried out for obtaining greater details over selected anomalies or indications; nor can this device eliminate the use of other geophysical methods entirely. The aerial instruments can only be effectively used for large scale or regional reconnaissance surveys where the structures of the geological formations sought for contain minerals of fairly high magnetic susceptibility.

There has been a good deal of publicity and advertisement regarding the new airborne magnetometer devices which may often tempt uninformed persons to contract for the use of the method at great expenditure without realizing the possibilities of, or need for, such surveys. In the advertisements and technical brochures comparisons of costs are sometimes given on the basis of what the ground surveys would cost and what large economies would result by the application of airborne surveys. While this comparison is true in appropriate cases, it should not be thought that the aeromagnetic surveys as such are cheap or inexpensive. For operations in countries outside U.S.A., the clients have to pay all the expenses of moving the plane, survey equipment, crews, etc., and meet the operational costs, living expenses, and all other inci-

dental charges of the working crews over and above the contracting fees. The overall costs may work up to many lakhs of rupees if such surveys were now taken up in India.

There is no doubt that there are some large areas of forests, inaccessible, hilly and mountainous tracts, deserts, and other blanketed or difficult ground in India where airborne magnetic surveys may bring to light some useful indications of iron ore bodies, or possible sub-surface structures favourable for finding oil. But will the location of iron ore in such blanketed tracts prove of any immediate benefit in India? The already known reserves of high grade ore in many parts of the country have not been tapped to any great extent. There is no point in applying a recent technique of exploration simply because it has been successful in U.S.A. The urgent necessity for location of iron ore deposits in that country is understandable as the very rapid growth of iron and steel production has brought in a fear of early exhaustion of their best and largest deposits.

Regarding the exploration for oil by aeromagnetic surveys, it must be understood that even in U.S.A. no outstanding discoveries or results in establishing new oil-producing fields have as yet been reported. It would appear that aeromagnetic surveys in India cannot command any priority, and there appears to be no urgency or need for

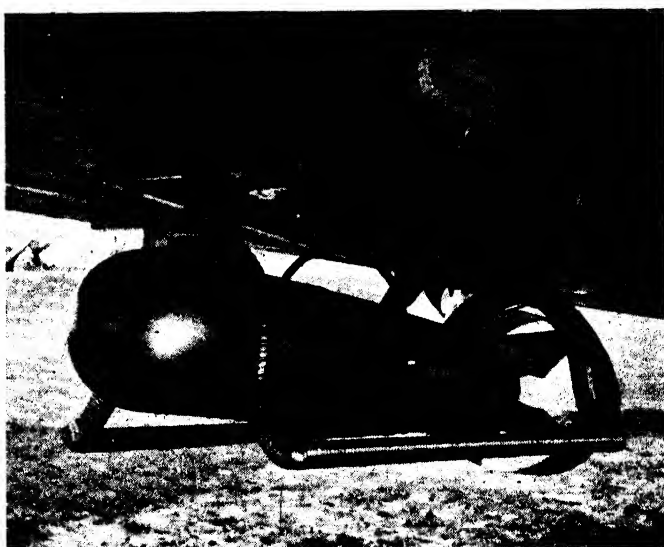


FIG. 2 — THE MAGNETOMETER PATENTED BY THE GULF OIL COMPANY AND USED BY THE AERO-SERVICE CORPORATION OF PHILADELPHIA.

(By courtesy of "Engineering & Mining Journal", McGraw-Hill Publication Co., New York.)

any of the Government or private agencies spending large sums of money on these surveys when the country needs all such capital, as it can spare, for the more feasible and urgently needed surveys by less expensive geophysical methods.

#### Prospecting for Radioactive Minerals

Dr. M. S. Krishnan has made a study of this subject and his valuable paper<sup>1</sup> on uranium gives an excellent summary of the present position of our known and potential sources; he also draws attention to the methods which can be employed in the testing of and exploration for radioactive minerals.

During the war some portable types of Geiger-Muller counters were designed in U.S.A. and used extensively in the search for radioactive ores. In the Yellow Knife and Eldorado areas of Canada, in portions of Colorado, Utah and other parts of U.S.A. — the famous sources from which most of the uranium ores were obtained for the atom bomb project — these portable radioactivity detectors have been used. There is strict secrecy attached to the several exploration programmes still actively going on in those countries.

The Geiger-Muller counters have been designed in portable form with self-contained batteries, tubes, headphones, and/or recording meters in a compact build for field use. The equipment is also convenient for use in

quarries, underground mine workings, tunnels, adits, shafts and other places. Several models of the portable counters have been placed on the market in U.S.A. and probably also in other countries now, and the cost of a single set would be about \$250. The operating expenses are small, and are mostly confined to the renewal of batteries.

In prospecting, this type of equipment could be worn on a shoulder sling, and the counter tube be adjusted to a convenient height above the ground. The operator walks at a slow pace and notes the counts registered on the meter in a given time, — say, per minute. There is usually a background count, due mainly to cosmic rays, which varies according to latitude and altitude; this count is also a characteristic of the particular counter tube employed. These "blanks" or background counts should always be determined over normal ground in the locality. Strong indications of the presence of radioactive minerals can be observed only when the background counts increase by 100 per cent or more. It is, however, necessary to bear in mind that thick overburden absorbs radiations from the underlying materials, and it is doubtful if veins containing valuable ore covered up by soil or debris to a thickness of more than 4' to 5' can at all be satisfactorily detected by this method. Major differences in the normal radioactive contents of several types of exposed rocks can be detected by this equipment if the background counts are small and remain otherwise undisturbed.

In India we may use the portable counters in tracts where geological conditions appear favourable for the occurrence of uranium and thorium minerals. Pegmatitic regions should be specially explored, the veins penetrating older schists and other metamorphosed rocks receiving a closer search. Faults and fissures sometimes have concentrations of radioactive substances and such zones may possibly lead to the discovery of likely sources of radioactive ore-body. It would also be worth while examining the wall rocks or faces in open

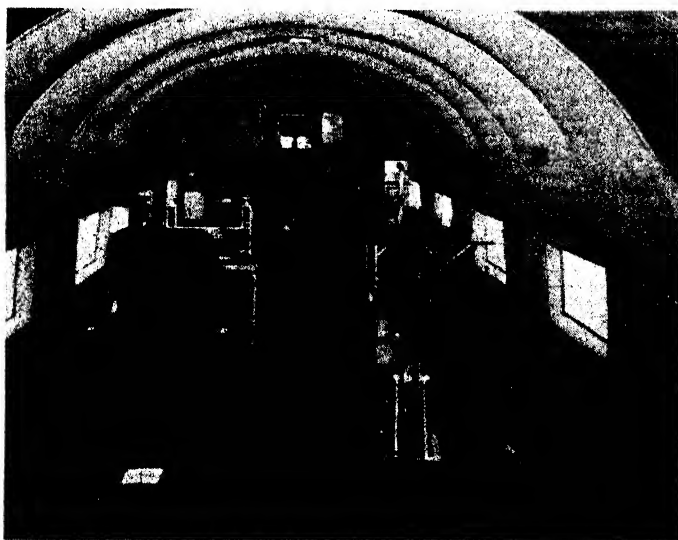


FIG. 3 — AN INTERIOR VIEW OF THE CABIN OF THE AÉROPLANE SHOWING THE INSTRUMENTS USED BY THE AERO-SERVICE CORPORATION OF PHILADELPHIA.

(By courtesy of "Engineering & Mining Journal", McGraw-Hill Publication Co., New York.)

cast and underground mine workings and quarries, and differentiating between rocks possessing varied degrees of radioactivity. Suitable counting equipments have been devised for measurement in wells and bore holes. There is also some scope for artificially exciting the radioactive properties of some of the minerals and measuring the secondary radiations. Such determinations may furnish diagnostic criteria for identifying and distinguishing certain of the rare minerals. A penetrating  $\gamma$ -ray has been discovered for beryllium<sup>2</sup>, and possibly with some further research a method may be developed by which beryl hidden in pegmatites can be detected.

The use of diamonds to detect atomic radiations has been recently suggested and the U.S. *National Bureau of Standards* have shown that diamonds are sensitive to  $\gamma$ -rays and that "Diamond Counters", like the G.M. counters, could be constructed to detect  $\gamma$ -ray radiations<sup>2</sup>.

While the foregoing lines of research and other possibilities of radioactive method should receive consideration of physicists and electronic engineers, a number of our geologists engaged in detailed mapping of pegmatitic regions should be furnished with portable Geiger-Muller counters and given training for using this apparatus in their field-work. However, the limitations of portable instruments as tools for prospecting must be realized. The detectability, as already pointed out, is rather poor for material covered by even comparatively thin overburden, and the method has practically no use on thickly blanketed ground. In spite of the extensive search made for uranium minerals in many parts of U.S.A. and Canada by a number of geologists belonging to the official surveys and private organizations, there have been no reports of any notable discoveries. In a review of the progress in mining geology

by Prof. L. C. Graton<sup>3</sup> published in the *A.I.M.E. Diamond Jubilee volume on Seventy-five years of progress in the mineral industry* it has been stated that "as far as yet disclosed, no spectacular additions to reserves of a grade hitherto minable resulted from the feverish and extended searches by the Allied Governments during war years"<sup>4</sup>.

Let us not entertain any unwarranted hopes of discovering large workable deposits of rich uranium ore. To think that marvellous results would flow from an extensive use of these portable detectors would serve no purpose. This instrument could be applied with the keenest appreciation of the factors involved in the method, if unnecessary disappointments are to be saved. It is probable that some areas may yield indications worth further attention for detailed



FIG. 4 — RADIOACTIVITY DETECTOR — THIS SHOWS THE PORTABLE GEIGER-MULLER COUNTER WHICH MEASURES THE INTENSITY OF  $\gamma$ -RAY RADIATIONS. THIS APPARATUS, COMPLETE AND SELF-CONTAINED WITH BATTERIES, COUNTING RATE METER, ETC., MEASURES 7"  $\times$  4 $\frac{1}{2}$ "  $\times$  3 $\frac{1}{2}$ ".

(By courtesy of Geophysical Instrument Co., Arlington, Virginia.)

prospecting. In this connection it is well to remember that any material containing lower than about 0.2 per cent of uranium cannot be regarded as usable ore, or stuff commercially feasible for processing in the extraction of the fissionable element<sup>4</sup>. The hopes entertained by some in relying upon large bodies of granites and other igneous rocks containing minute quantities of uranium and thorium as potential sources for extraction of these elements appear to have no practical or economic basis.

### Seismic Methods of Prospecting

During the Great War I, a sound-ranging technique was developed to locate the position of enemy guns. The apparatus enabled one to measure the time taken for sound waves from the guns to travel to distant points. Soon after, attempts were made to use this principle for determining sub-surface geological structures. Seismic prospecting is based on a study of the elastic sound waves generated by a blast of explosive in the ground. The waves are picked up by delicate seismometers (or geophones as they are called) placed at different spots, and the records of the times of arrival of the waves are carefully investi-

gated. From these measurements it is possible to infer the nature of strata through which the waves have been transmitted and calculate the depths to geological discontinuities. Two types of investigation are possible in this method: they are described as the refraction and the reflection shooting methods. The difference between the two consists in the nature of the waves (refracted or reflected) whose times of arrival are studied. In the refraction method, travel-time data are obtained for those waves which have been *refracted* at the boundaries of geological formations or strata possessing different elastic constants (or density). In the reflection method, observations are made of the time of arrival of the waves which have been *reflected* from the sub-surface horizons possessing distinctive elastic constants. The two methods differ in numerous practical details and theoretical considerations.

Reflection seismic surveys are the most popular ones in recent years in U.S.A. for the exploration of oil. Some of the reports show that the results of actual drilling have confirmed the oil-bearing structures accurately to depths as large as about 15,000' below the surface. A lot of development both in instrumental design and field technique of exploration has taken

place and it is needless to point out the extraordinary successes which have attended this method in the location of geological structure favourable for finding oil.

Geophysical prospecting for petroleum is a most highly specialized technique involving the use of elaborate equipment and highly trained personnel. Enormous sums of money are required for the instruments, field equipment, laboratory, personnel, operating charges and other investments. The geological search for oil is an "industry" by itself in U.S.A. and Canada, and it has been almost entirely carried out by private organizations who command large resources of finance from their various undertakings yielding substantial profits. In 1945 the oil business in U.S.A. spent over \$55,000,000 in geophysical prospecting

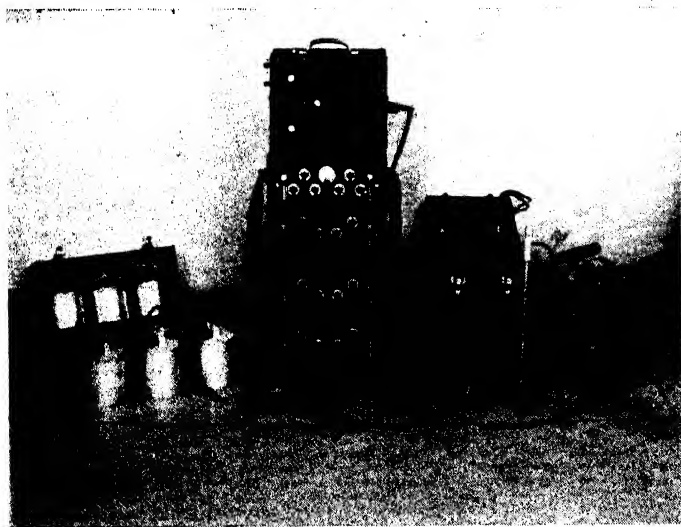


FIG. 5—PORTABLE SEISMIC EQUIPMENT SUITABLE FOR REFLECTION SHOOTING. THE SIX CYLINDRICAL UNITS TO THE LEFT ARE THE GEOPHONES WHICH PICK UP THE PULSATIONS FROM THE GROUND. THE SMALL BOX AT THE EXTREME RIGHT IS THE "SHOOTING BOX"—A CONTRIVANCE TO ELECTRICALLY CHARGE THE EXPLOSIVES. THE 6-TRACE CAMERA AND OTHER CONNECTED PARTS ARE CONTAINED IN THE PANEL-CASINGS SHOWN IN THE CENTRE.

(By courtesy of Heiland Research Corporation, Denver, Colorado.)



and employed more than 1,000 professional geophysicists. Even so, the oil industry may be said to have used only about 3 per cent of the value of the oil production on geophysical prospecting<sup>5</sup>. The oil companies in India are also probably spending several lakhs of rupees on geophysical surveys, although it is not known what proportion this expenditure bears to the total value of their oil production. Legislators and others in India are demanding that the Government should conduct geophysical surveys for locating more oil. Dr. G. Dessau in a recent paper entitled *Past & Future of Exploration Geophysics in India*<sup>6</sup> has, among other questions, already discussed the pros and cons of Government agencies in India taking up such geophysical surveys.

Whatever may be the position with regard to the large-scale surveys for oil, there should be no doubt at all that some seismic prospecting programmes on a limited scale can be undertaken by Government agencies as is being done by the *U.S. Geological Survey*. I would specially stress the need for obtaining portable seismic equipment, several types of which have been introduced in recent years. Some of these portable units which have been placed in the market are compact and well constructed and are likely to be found quite suitable for operations in India.

In particular, the *Technical Instrument Company* (Houston, Texas) have put up a unitized portable seismograph which weighs only about 55 lb. It has a 24-trace camera with mirror galvanometers (electrically damped moving coil type) arranged for dual reading of 12 tracers with 12 amplifiers and a neat central panel, having a 30-cycle oscillator for checking phase and balancing amplifiers. The tuning fork has a vacuum tube drive and is magnetically coupled to the timing motor. One 6-volt automobile battery is the only power required for operating both the camera and the amplifiers. Together with this unitized seismograph, companion equipment such as geophones, shooting box, telephones, cable tester, etc., are also available. The galvanometers and amplifiers are replaceable. The manufacturers guarantee the set for satisfactory operation for one year. The cost is rather high, about \$12,000 for the basic unit. A number of oil companies have such units in operation and it is reported that the results obtained with the portable sets are equal

to the performance of the conventional heavy equipment.

The *Heiland Research Corporation* of Denver, Colorado, have also a 6-trace portable equipment which is said to be excellent. The price is approximately \$6,000.

The *Geophysical Instrument Company* of Arlington, Virginia, have placed on the market an ingenious portable seismograph designated "Shepard Type" (named after the original designer Mr. E. R. Shepard, Geophysicist of the *U.S. Army Corps of Engineers*). This instrument has a 3-trace camera with a tuning-fork device for obtaining the time lines. The firing apparatus is included within the recorder unit itself, thus eliminating the need for a separate blasting apparatus. The whole equipment with all the accessories weighs 125 lb. and the cost is about \$5,000. This type is suitable only for shallow refraction-shooting problems.

The *U.S. Army Corps of Engineers* and *U.S. Bureau of Public Roads* have employed the above type of portable seismographs in many of their projects and obtained useful results. Thanks to Mr. E. R. Shepard, the author was able to study in some detail the geophysical work for the Lehigh Valley Scheme near White Haven in East Pennsylvania State. The main object of the Scheme, which was under investigation by the *U.S. Army Corps of Engineers*, was the construction of a high dam across the Lehigh river for flood control and water power development. The geophysical investigation by Mr. Shepard consisted of the examination of alternative sites for the dam. The depths to bed-rock at various spots were determined by using the portable seismograph and the results obtained were satisfactory, facilitating a rapid evaluation of the relative merits of the sites.

Another seismic investigation conducted by the Geophysics Section of the *University of Toronto* in connection with a water supply scheme near North York township in Ontario Province was studied by the writer. This scheme was to sink a number of tube wells and obtain water for farm areas and industrial plants to be set up in that township. The seismic survey was carried out to outline the details in the bed-rock topography hidden by overburden (glacial fill) and select the "deep leads" or underground channels in bed-rock from which better supplies of water could be tapped. The whole project was sponsored by the Ontario Provincial



Government. The seismic data obtained revealed some favourable spots for sinking wells. Many such investigations are going on in America and elsewhere, and we in India ought to apply the seismic technique in many of our projects.

Another recent investigation by Mr. W. E. Grasham of the *Canadian National Research Council*, Ottawa, with acoustic echo-ranging devices may be mentioned here. Attempts have been made to study the applications in geophysics of sonic echo-ranging methods<sup>7</sup>. For measurements of the depth of overburden, standard seismic reflection technique has been modified to use higher frequencies of the order of 1,000 cycles per second with small explosions providing the source of sound.

Yet another recent development of considerable engineering interest is the design of special geophones by the *U.S. Bureau of Mines* in the investigation of micro-seismic impulses set up in rock-faces or pillars in mining and civil engineering constructions. A number of papers by Dr. Leonard A. Obert and Dr. Earnest A. Hodgson of Canada have been published on this topic<sup>8</sup>. By the courtesy of Dr. Obert, senior physicist of the *U.S. Bureau of Mines*, the author was able to gather some details of the method. The equipment built up in this laboratory has devices for both listening to and recording the sub-audible snappings or "micro-seisms" as they are called, which are assumed to be associated with rocks under high pressures approaching their crushing strength. For any given specimen of rock of suitable dimensions, the critical rock pressure can be detected by this equipment and the technique is being extended to the study of rock-bursts in deep underground mines.

In Canada a long series of researches under the direction of Dr. Earnest A. Hodgson, Chief of the Division of Seismology, *Dominion Observatory* at Ottawa, have been carried out at Lake Shore, Kirkland Lake, and other deep gold-mines in the Ontario Province. Although the results of most of these researches have not yet definitely established a technique for the prediction of rock-burst in underground mines, it has been shown that methods could be worked out to indicate whether or not a given stope is under critical pressure at any given time. Maybe, further researches would eventually succeed in improving this method to predict rock-bursts.

In India, too, our engineers may welcome the assistance obtainable by using the geophysical methods, where applicable, in solving problems connected with the investigation of the vast variety of projects. Electrical and seismic methods offer definite scope for aiding the engineers in the investigation of dam sites, highways, railroads, tunnels, aqueducts, pipe lines, channels, cut-and-fill excavations and other allied works and underground water supply problems.

### Gravitational Methods

The application of gravitational methods as an aid to prospecting for petroleum in U.S.A. started more or less about the time of World War I. With the use of the Eotvos torsion balance, geological structures such as anticlines and domes having cores of material of different densities were located. The first oil pool said to have been discovered by geophysical methods in America during 1926 was by the gravitational method. However, owing to the high cost of exploration with the torsion balance little progress was made in the beginning.

The development of and improvements to the gravimeters, however, have soon brought about a wider use of the gravitational method, and at present quite a large number of discoveries are attributed to its use. While the torsion balance measures the horizontal gradient of gravity and the differential curvature, the gravimeter measures directly the relative differences in the force of gravity by "weighing" the same object from point to point. The usually observed changes in weight are exceedingly small — of the order of one part in ten thousand — or only about one ten-millionth part of the value of gravity. The gravimeters are designed to measure such minute variations in the weights of an object as "weighed" at different locations.

Many types of new gravimeters — light and portable and capable of making accurate measurements — have come into the market. Some of these are suitable for use in the investigations for deposits of minerals of high density and also for location of structures like faults and flexures. An attempt has been made in India by Dr. B. L. Gulatee, on an experimental basis, to trace the manganese ore deposits concealed under overburden by the gravimetric method. It is also learnt that the *Survey of India* have already obtained a Frost Gravimeter from

U.S.A., and that some surveys have also been undertaken in the Raniganj coalfield.

There is no doubt that we could successfully apply the gravimetric method in a few of our investigations — both reconnaissance and exploratory surveys — and delineate known ore bodies and outline certain structures hidden by overburden. Trained personnel to use the gravimeters and interpret the results would be available soon when a few of our young men now undergoing regular courses of geophysical study abroad return to India.

### Electrical & Magnetic Prospecting

The later developments in the electrical and magnetic methods have resulted in the design of many types of specialized instruments and new techniques. Improvements and variations in the techniques have enabled a more extended use of these methods. In the resistivity methods, the improved Gish-Rooney apparatus holds a prominent position, although the use of vacuum tube voltmeter equipment has been increasing in certain fields. Continuous or semi-continuous resistivity measuring devices with mobile electrodes and oscillograph records mounted on trucks have been introduced. A new variation of the resistivity technique suited to investigation by means of measurements in boreholes in the mining field has also been worked out.

Some new designs and types of non-polarizable electrodes have been introduced for measurements in the self-potential method. The electrical transient and the telluric current methods are an innovation. In the high frequency and electromagnetic methods, too, some new instruments have been introduced although various factors appear to have restricted a more general use of the inductive methods. On the whole, the tendency has been more or less a reversion to the older electrical methods like the earth resistivity and spontaneous polarization methods in the mining field.

The Askania magnetometers are now exceedingly difficult to procure and several other makes have come into the field. Especially the Ruska magnetometers (*Ruska Instrument Corporation*, Houston, Texas) are said to be an improvement on the Schmidt type of magnetic field balance. In the older forms of the Schmidt magnetic balances, there were some operating difficulties and the delicate instruments had to be handled with great care and skill only by experienced

operators; otherwise, servicing the instruments was a hard job. The new Ruska magnetometers are claimed to remain accurate for long periods and the operator himself can make most of the necessary adjustments rapidly in the field. Also, the *Ruska Corporation* have put forward new types of base-station recording magnetometers, which without the need of attendants, dark tents and extensive set-ups, give a complete record of the diurnal variations, time and temperature. The recording instrument also permits direct visual observations at any time without disturbing the record which is being made.

The *Geophysical Instrument Company*, Arlington, Virginia, have also developed a magnetometer following the lines of the older Schmidt type and incorporating improvements in the construction and design of the knife-edge, etc. *Sharpe Instrument Company* of Toronto, in Canada, have also recently put on market some magnetometers. Dr. Hans Lundberg's organizations are developing magnetometers for continuous recording on land surveys or over-water surveys.

While considerable progress has been made in instruments and techniques, there has also been a better understanding in the interpretation of the survey data and many useful results have followed the application of the electrical and magnetic methods in the mining field and civil engineering investigations. A few of the outstanding results obtained in the mining field in Canada are given below.

Not many years ago, the practical value of geophysical prospecting methods for the mining field was doubted by many. In Canada they were taken sceptically at first by the mining industry. But today, following a number of rather spectacular successes, the methods have gained a high reputation. The mining industry there spends thousands of dollars on geophysical survey programmes. There has also been a tendency on the part of some of the mining companies to have their own staff for doing geophysical work.

In the Sudbury field large deposits of nickeliferous pyrrhotite have been discovered in recent years by geophysical methods under careful geological supervision<sup>9</sup>. The discovery of ore in Levack township, Falconbridge area, has been one of the most outstanding successes for the magnetic method. The property has developed into an important mine, producing about 10,000 tons of

nickel and about 5,000 tons of copper annually. It is reported to have reserves of nearly 8 million tons of sulphide ore.

In the most prominent gold-fields of Canada—the Porcupine, Kirkland Lake, and Lake Shore mines, geophysical methods have been employed for gaining information on the structural features.

In the comparatively recent mining camps near Noranda and Bourlamaque in Quebec Province, electrical and magnetic prospecting methods have been very actively employed. The *Queмонт Mining Corporation*, who had their properties adjacent to the most productive mine known as the *Horne Mine of Noranda Mines Ltd.*, got their lands geophysically surveyed in 1944. No outcrops of rocks are available in the area and 20 years ago a 900' shaft had been sunk on the property and a large amount of lateral development carried out, resulting in the location of negligible quantities of ore and expenditure of over half a million dollars. The property was almost idle till 1944 when a few magnetometer surveys were carried out by *Messrs Koulomzine, Geoffrey, Brossard & Company* of Valdor, Quebec<sup>10</sup>. The first drilling which followed their geophysical work had not produced particularly encouraging results, but in 1945 when the diamond drilling work beneath Osisko Lake was carried out, valuable sulphide ore bodies were intersected. These subsequently have been developed by extensive drilling, shafting and drifting and the later developments have shown a reserve of over 6 million tons of workable sulphide ores having 1.58 per cent of copper and 0.18 oz. of gold per ton.

In the Valdor area (Quebec Province) in the East Sullivan Property near Bourlamaque township, intensive magnetic surveys carried out by Dr. T. Koulomzine during 1940 have been responsible for successfully tracing the hidden contacts between the older greenstones and granodiorite batholiths—that is, zones which from experience gained in other parts were known to contain auriferous lodes<sup>11</sup>. At least two previously unsuspected plugs of granodiorite which are hidden by thick glacial overburden or waters of the lake were discovered. The subsequent drilling has disclosed important auriferous lodes. The *East Sullivan Mines Ltd.* are reported to have carried out over 50,000' of diamond drilling and developed ore bodies which are now producing annually about 1½ million dollars worth of gold.

In the Lynn Lake area in Northern Manitoba, the geophysical surveys are said to have shown some two or three hundred anomalies and detailed investigations are being carried on to assess their values<sup>12</sup>. By the courtesy of Mr. J. T. Randell of the *Geo-Technical Development Company* of Valdor, who had conducted electrical and magnetic surveys over some of the properties of the *Sherritt Gordon Mines Ltd.* and the *Hododoo Mines Ltd.* during July 1947, the author gathered some details of the nature of the geophysical investigations in that region. It is reported that the drilling up to the close of 1947 by the *Sherritt Gordon Mines Ltd.* has revealed 6.8 million tons of sulphide ore averaging 1.74 per cent nickel and 0.75 per cent of copper. Particularly, one of the anomalies there has showed up nearly 2 million tons of sulphide ore containing 3.28 per cent nickel and 1.76 per cent copper. All recent reports<sup>13</sup> show that in the Lynn Lake area there is going to be eventually a new mining district with several mines producing nickel and copper.

It is remarkable that the three important economically workable bodies of ore discovered in recent years in Canada (in the Quemont area near Noranda; in East Sullivan near Valdor; and in the Lynn Lake area in Manitoba) are all directly attributable to the successes in the application of magnetic and electrical methods<sup>14</sup>.

#### Geological Studies in Geophysical Programmes

Perhaps one of the notable features in the recent advances in geophysical exploration is the increasing appreciation of the importance of geological studies. In the earlier stages of the development of exploration geophysics hopes might have been entertained that instruments and methods could be devised to grind out mechanically answers to our problems. But experience has shown that the mere physical measurements relative to the determination of the electrical, magnetic or gravimetric anomalies do not have much meaning or significance in themselves, and that geophysical methods cannot offer any direct indications of ore or other mineral substances under the conditions in which most mineral deposits occur in nature. The geophysical data can assist the geologist and the mining engineer in their geological deductions in cases where there is paucity of outcrops for direct observation. In many of the geophysical papers which have

been published in recent years, the importance of a thorough appreciation of the geological factors in interpreting the geophysical data has been emphasized. A large-scale geophysical indication connected with the structures in the oil-field is detected by suitable technique and interpreted definitely. But most of the ore deposits form very much smaller geological features and do not yield indications on a large or regional scale. The geophysical anomalies in the latter cases are usually of a localized character and are often complicated by a number of factors depending upon the local geological conditions. A proper evaluation of the geophysical anomalies can be made only if the geological probabilities are properly appreciated and well understood. This naturally calls in for a great deal of attention to geology. Dr. C. A. Heiland and Wantland in one of their papers have remarked that "it is regrettable that the word (Geophysics) is not long enough to get a little more of geology into it"<sup>15</sup>. Let me cite one very interesting account by E. Poldini who has detailed some considerations relative to geophysical interpretation<sup>16</sup>. A problem in electrical prospecting solvable graphically was submitted to seven persons — geophysicists, geologists, engineers and technicians — in order to determine the extent to which a subterranean topography or relief could be reconstructed on the basis of electrical measurements. Each person was given only a blank sheet with values of apparent resistivity over a net-work of stations. The invitation was to draw the corresponding contour lines of equal resistivity and to give a geological interpretation. The seven contour maps offered as solutions and a map of the true relief when compared are reported to have shown that three maps drawn by competent geologists approached the correct solution closely whereas the other maps drawn by engineers, technicians and one geologist deviated from it widely.

### Conclusion

The foregoing account of the recent progress in exploration geophysics is by no means exhaustive, and it should be mentioned that there has been a geo-chemical approach to many prospecting programmes, especially in the search for ores. This method perhaps began about the year 1937 in Europe taking into account the enrichments denoting concentrations of some of

the trace elements in topmost layers of soils by processes of evaporation and filtration incidental to the growth of plants. The geo-chemical unit of the U.S. *Geological Survey*, under Mr. Hawkes, has made many extensive tests and experiments to ascertain the possibilities for conducting field analyses of water, plants, residual and alluvial soil and glacial till. Their results have shown that with simple equipment many useful analyses may be made to assist in the search for ore deposits.

From the increasing volume of literature which is being published, it is clear that numerous geophysical and allied methods are extensively used in many countries — in Europe, Australia and Africa. Improved designs of geophysical potentiometers, megger earth testers, magnetometers and seismographs have been put on the market in England. There has been considerable progress in Sweden too — one of the countries which was the earliest to use geophysical methods. Special types of portable seismic equipment and the "Norgaard Gravimeters" have been designed by the *Electrical Prospecting Company of Stockholm*, Sweden. The electromagnetic "Loop-Frame" and the "Boliden" gravimeters developed by the *Boliden Mining Company of Sweden* have been used in Europe. These notable developments have contributed to bring in a greater appreciation of the usefulness of the geophysical methods.

In Asia the progress in the application of geophysics has not been anything comparable to that achieved even in Australia or Africa. There is a vast scope for geophysical exploration in many parts of Asia. Private capital seems particularly shy to come forward to sponsor any important schemes for geophysical exploration. The *Burmah Oil Company* and the *Anglo-Iranian Oil Company* are the only private organizations who are sponsoring geophysical surveys in India, Pakistan and Burma. Much of the initiative for further progress, at any rate, for some time to come will be the responsibility of Government departments.

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## Chemical Control for Power Alcohol Distilleries—a Scheme

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ONE of the urgent needs of the power alcohol industry is to maximize the output and bring down the cost of production of power alcohol. This can be realized only if the control methods are all standardized and brought under an unified scheme. Among the different items making up the cost of power alcohol, viz. the expenses of raw materials, steam, labour, maintenance of machinery, overhead charges, depreciation, etc., only the first two items come under the scope of technical control. A strict control of the processes leads to the highest possible recovery of alcohol from a raw material like molasses. Similarly, a careful accounting of the heat balance in the distillation plant leads to a saving of fuel for the boilers.

A standardized system to achieve this two-fold control is particularly lacking in the alcohol industry. We have, in the sugar industry, a perfect system of chemical control and a uniform scheme for reporting factory data, although on the side of steam economy, much is still desired. It is desirable that on the same lines as in the sugar industry, a scheme for chemical control is worked out for power alcohol also.

The present article deals with a proposed system of chemical control in the distillery

and a uniform method for reporting factory data.

Any system of chemical control has a threefold objective :

(1) To collect working figures and data at every step of the manufacturing process with a view to exercise efficient control of this step.

(2) To provide accurate figures for preparing periodical run reports.

(3) To provide a uniform basis for all factories in the industry for mutual exchange and comparison of data and efficiency figures.

Having the above objectives in view, the present scheme gives precise definitions for all the technical terms and expressions that are proposed to be used in the power alcohol industry. While formulating such definitions, importance is given more to the precision and clarity than to agreement with the correct scientific meanings. For instance, it is sufficient for the purpose of chemical control to define the strength of absolute alcohol as 99.5 per cent and above, as has been done in the pharmaceutical industry, although the rigorous scientific definition applies only to 100 per cent alcohol. In a similar manner we want only workable and precise definitions for the terms "sugars",

"fermentable sugars", and so on. The next requirement is to work out expressions and control formulae. In the case of the power alcohol industry it has been found necessary to define the units on which such expressions are based. In the parallel case of sugar manufacture, sugar is present as such in the raw material—cane or beet. Therefore, it provides an easy basis for expressing the quantities as percentages of the raw material. However, as alcohol is manufactured from the sugars in the raw material, a simple percentage basis is not very convenient to have. According to usage, "recovery.", for instance, has to be expressed in imperial gallons per maund or ton of raw material. Difference of opinion is bound to arise when taking the basis of any such system of control. Similarly, there is a pronounced divergence of views on the point of adopting imperial gallon or the London proof gallon as the unit for measuring quantities of spirit.

In the system to be described herein, preference has been given to usage in the trade rather than to what may be a strictly scientific procedure. The Bengal *maund* has been taken as the unit of weight, and the London proof gallon as the unit of volume of spirits.

The third requisite is a suitable *pro forma* for the periodical run reports which could be recommended uniformly for the factories. This has been proposed with a view to making it as general as possible, but, whenever it is adopted in any particular factory, certain items, which are necessary for the special working conditions there, have to be added.

While reporting the time account, it is desirable to differentiate stoppages of process which have unavoidably taken place from those stoppages which could have been prevented in the normal course.

#### General Definitions

1. *Molasses* — The mother liquor separated from sugar crystals and sent to the distillery for use as raw material for processing and producing alcohol.
2. *Sugars* — The total of the mono and polysaccharides in a product capable of reducing alkaline Fehling's solution and expressed as "invert sugar".
3. *Unfermentable sugars* — Sugars remaining over after the fermentation of a product by a pure yeast culture under optimum conditions.
4. *Fermentable sugars* — "Sugars" — "Unfermentable sugars".
5. *Brix* — Density of a product expressed on Brix densimetric scale and taken as percentage of solid matter present in the product.
6. *Alcohol* — The pure chemical known as ethyl alcohol or ethanol and represented by the formula  $C_2H_5O$ .
7. *Spirit* — A mixture of alcohol with water in any proportion.
8. *Proof spirit* — Such spirit as would weigh at 51°F. 12 to 13 times an equal volume of pure water also at 51°F.
9. *Degrees over-proof (O.P.)* — The number of degrees over-proof of any spirit is the excess number of volumes of proof spirit which can be obtained by diluting 100 volumes of the spirit. Alcohol is "75.35° O.P."
10. *Degrees under-proof (U.P.)* — The number of degrees under-proof of any spirit is the difference between 100 and the number of volumes of proof spirit contained in 100 volumes of the spirit.
11. *Degree gravity* — The degree gravity of any product expresses the density by taking the first three decimal figures of its specific gravity.
12. *Wort* — The solution of molasses prepared for alcoholic fermentation.
13. "*Pitch*" or "*Bub*" is the yeast slurry obtained from "wort" or otherwise for inoculating the main fermentation vats.
14. *Wash* — The solution of molasses in any stage of alcoholic fermentation.
15. *Sludge* — Sediment obtained from the bottom of fermentation vats.
16. *Spent wash* — The waste effluent from the bottom of wash distillation column.
17. *Spent lees* — The waste effluent from the exhausting section of the alcohol concentrating column.
18. *Rectified spirit* — The concentrated alcohol drawn from the rectification column.
19. *Dehydrated alcohol* — The product obtained by further elimination of water from the alcohol-water azeotrope.
20. *Absolute alcohol* — Any spirit containing 99.5 per cent or more of alcohol by volume.
21. *Denatured spirit* — Any spirit rendered unfit for potable purposes by the addition of such substances, and in such proportion, as may be prescribed statutorily.
22. *Degrees attenuation* — Fall in the degrees gravity between two stages of alcoholic fermentation.

23. *Conversion factor* — Designated by the symbol "*f*" is the maximum weight of alcohol obtainable by the fermentation of one unit weight of fermentable sugars. "*f*" =  $0.5111 \times 0.9459 = 0.4845$ .

#### Technical Terms & Control Formulae

*Direct Determinations* — The following quantities are determined directly by laboratory methods:

1. *Brix molasses* — By the use of "Brix spindle" or any other densimetric device. Brix molasses is taken as the per cent weight of solid matter in molasses.
2. *Sugars per cent molasses* — By inverting a composite sample and determining the reducing sugars by the usual Fehling's method. The result is expressed as "invert sugar".
3. *Unfermentable sugars per cent molasses* — By fermenting a sample of molasses with an approved culture of pure yeast and determining the unfermented sugar by the usual Fehling's method.
4. *Fermentable sugars per cent molasses* — The difference between (2) and (3) will give fermentable sugars per cent molasses, and is checked approximately by the distilled alcohol during this estimation.
5. *Gravity wort (initial)* — By a hydrometer or any other suitable densimetric device. The net initial gravity of the wort, in any set-up, is obtained by correcting for the effect of "pitching". The corrected initial gravity is used in all calculations for purposes of high accuracy.
- 5a. "Brix wort" can be found by reference to Brix and specific gravity tables.
6. *Sugars per cent wort* — By inverting a composite sample and determining the reducing sugars by the usual Fehling's method. The result is expressed as invert sugar. For "deduction method" for sugar per cent wort, *vide* "Deduced Expressions III".
7. *Gravity wash (final)* — By hydrometer or any other suitable densimetric device.
- 7a. "Brix wash" can be found by reference to Brix and specific gravity tables.
8. *Residual sugar per cent wash (sugar per cent wash)* — By determining the reducing sugars by the usual Fehling's method. The result is expressed as invert sugar.

9. *Alcohol per cent wash (by vol.)* — By laboratory distillation of a composite sample of wash.
10. *Gravity spent wash* — *Vide* No. 7.
11. *Alcohol per cent spent wash (by vol.)* — By single or multiple distillation of a composite sample and determining alcohol by densimeter or chemical analysis.
12. *Alcohol per cent spent lees (by vol.)* — *Vide* No. 11.
13. *Alcohol per cent rectified spirit (by vol.)* — A hydrometer or any other densimetric device. The strength is also expressed in proof degrees.
14. *Alcohol per cent dehydrated alcohol (by vol.)* — *Vide* No. 13.
15. *Alcohol per cent any spirit (by vol.)* — *Vide* No. 13.

The following quantities are determined from direct measurements or are calculated:

16. *Molasses processed (Number of maunds of molasses diluted and converted into wort)* — By weighing or determining the volume.

The following expression can be employed for the indirect method of calculating the molasses processed:

$$\frac{\text{Wort made (gal.)} \times 10 \times \text{sp. gr.}}{82.27} \times \frac{\text{Molasses \% wort}}{100}$$

17. *Distilled wash* — in gal.
18. *Sludge per cent wash* — in gal.
19. *Pitch ratio* is the proportion of molasses used in preparing the pitch to the molasses processed.
20. *Spent wash* can be calculated sufficiently approximately by the relation: wash + steam = spentwash + equilibrium vapour = wash + steam - equilibrium vapour. The steam admitted to the stripping column can be assumed as a constant figure per unit volume of spirit made.
- The composition of equilibrium vapour and hence the quantity can be found by reference to standard tables giving the vapour compositions corresponding to the alcohol concentrations in the wash.
21. *Spent lees* — Either by direct measurement or from the composition of the equilibrium vapour and the quantity of spirit drawn as product.
22. *Spirits made* are measured by the standard methods of taking the dip readings in calibrated receiving tanks.
23. *Proof strength of spirit* — By the standard Sike's hydrometer.
24. *Alcohol made, in gal., from 22 and 23.*

**Deduced Expressions**

NOTE: The numerals within brackets indicate the "Direct Determination".

The following quantities and expressions are deduced from "Definitions" and "Terms":

I. *Possible recovery*—(The number of gal. of spirit available from the fermentation of one maund of raw material under optimum conditions) = fermentable sugars per cent molasses (4)  $\times 0.08792$ .

II. *Molasses per cent wort*

$$= \frac{\text{Brix wort (5a)}}{\text{Brix molasses (1)}} \times 100$$

III. *Sugars per cent wort* (6) = Sugars per cent molasses (2)  $\times$  molasses per cent wort (II)  $- 100$

$$= \text{Sugars molasses (2)} \times \frac{\text{Brix wort (5a)}}{\text{Brix molasses (1)}}$$

This formula may be used for the deduction of sugars per cent wort as an alternative to the direct determination (6).

IV. *Fermentable sugar per cent wort* = Fermentable sugars

$$\text{molasses (4)} \times \frac{\text{Brix wort (5a)}}{\text{Brix molasses (1)}}$$

V. *Fermentation recovery*

$$= \frac{\text{Alcohol \% wash (9)}}{\text{Possible recovery (I)}}$$

VI. *Fermentation efficiency*

$$= \frac{\text{Fermentation recovery (V)}}{\text{Possible recovery (I)}} \times 100$$

When the "sludge" in fermentation vats are wasted, we have the following expressions — VII and VIII.

VII. *Fermentation house recovery*

$$= \text{Fermentation recovery, (V)} \left( 1 - \frac{\text{sludge \% wash}}{100} \right)$$

VIII. *Fermentation house efficiency*

$$= \frac{\text{Fermentation house recovery (VII)}}{\text{Possible recovery (I)}} \times 100$$

IX. *Distillation house efficiency* — (Alcohol distilled per cent alcohol present in wash)

$$= \frac{\text{Alcohol made (24)}}{\text{Distilled wash (17)} \times \text{Alcohol wash (9)}} \times 100$$

X. *Overall recovery* — (The number of gal. of spirit recovered in the distilled products per maund of molasses).

XI. *Overall efficiency*

$$= \frac{\text{Overall recovery (X)}}{\text{Possible recovery (I)}} \times 100$$

**Loss Account**

The difference between the "possible recovery" and the "overall recovery" represents the "overall loss".

On the analogy of the methods of sugar industry it is possible to account for this "overall loss" under different heads. The loss in a distillery occurs in the following ways:

1. *Sludge loss* — No. of gal. lost per maund of molasses processed in the sludge from the bottom of the fermentation vats.
2. *Spent wash loss* — No. of gal. lost per maund of molasses processed in the spent wash.
3. *Spent lees loss* — No. of gal. lost per maund of molasses processed in the spent lees.
4. *Undetermined loss* — Due to evaporation from the fermenters, consumption of sugars for the building of yeast cells, side reaction, production of by-products like fusel oil and acetic acid, evaporation from hot condensers, leaks and spillage.

It is desirable for every distillery to prepare a loss account under the above four items. Item Nos. 1, 2 and 3 can be calculated, and item No. 4 can be found by difference.

**PERIODICAL REPORT**

Name of distillery ..... Period.....  
Process used..... Date.....

**(a) TIME ACCOUNT**

	Month	Date
1. No. of period hours		
2. No. of available hours		
3. No. of run hours		
4. Output per 24 period hours		
5. Output per 24 available hours		
6. Output per 24 run hours		

**NOTE:**

- (1) Period hours are total hours in the period.
- (2) Available hours are period hours less stoppages due to causes beyond control like molasses and fuel shortage and full warehouse.
- (3) Run hours are available hours less stoppages within control like cleaning, repairs, and water, steam and power failure.



## (b) QUANTITIES CONSUMED

	Month	Date
1. Molasses (maunds)		
2. Ammonium sulphate "		
3. Sulphuric acid "		
4. Phosphate "		
5. Benzene (gal.)		
6. Coal (maunds)		

## (c) PRODUCTS —

## QUANTITIES MADE

*Direct*

1. Wash (gal.)
2. Absolute alcohol "
3. Other spirits "
4. Fusel oil (lb.)

*Indirect*

1. Power alcohol (gal.)
2. Denatured spirit "
3. Any other spirit "

## (d) ANALYSIS

1. Brix molasses
2. Sugar % molasses
3. Fermentable sugar %  
molasses
4. Gravity wort (corr.  
initial)
5. Gravity wash (final)
6. (Residual) Sugar wash  
(final)
7. Alcohol % wash (by vol.)
8. Alcohol % spent wash  
(by vol.)

9. Alcohol % spent lees  
(by vol.)

10. Strength of absolute alcohol, O.P.
11. Strength of other spirits, O.P. or U.P.

## (e) EFFICIENCY FIGURES

1. Possible recovery (gal.)
  2. Fermentation house recovery (per maund molasses)
  3. Overall recovery (per maund molasses)
  4. Losses (gal. per maund of molasses)
- Sludge  
Spent wash  
Spent lees  
Undetermined  
Total
5. Fermentation efficiency %
  6. Distillation efficiency %
  7. Overall efficiency %

## (f) PROPORTIONS

1. Ammonium sulphate  
(% molasses)
2. Sulphuric acid "
3. Phosphates
4. Benzene (gal./1,000  
gal. abs. alcohol)
5. Coal (md./gal. alcohol)

## Patent Rights & Co-operative Research

CO-OPERATION BETWEEN INDUSTRY AND public-financed research institutions is a matter of vital importance for the furtherance of research effort. There is scope for greater financial support by industry in the promotion of research at such institutions. The assistance need not arise out of philanthropic considerations alone and may be quite in keeping with the fundamental basis of industry which is to make profit.

A mutually satisfactory arrangement consists in the exchange of patent rights for financial aid from industry. There are many fields of research which are likely to yield results having a high degree of patentability

and a reasonable chance of profit. Interested industries may give direct financial support to such research in universities and like institutions in return for exclusive patent rights to the results of investigations. The exchange of patent rights for financial aid provides a flexible arrangement as the details can be varied to suit each case.

The suggested arrangement would leave the universities and research institutions to concentrate on research and training, while industry may look to the business of obtaining, developing and exploiting the patents.

R. B. P.

## "The Blending of Coals for Coking"\*

THE manufacture of coke is of basic importance to the production of iron and steel, and, for this reason alone, it has a special claim to consideration in schemes for the industrial development of any country. It is well known that, although India has large supplies of iron ore of excellent quality, the reserves of good coking coal are limited; and it is, therefore, natural that attention has been focussed on the problem of stretching our coal reserves for coking purposes.

Towards the end of 1940, a scheme for research on the blending and coking of coals was initiated by the *Board of Scientific & Industrial Research* under the joint auspices of the Government of India and of the *Tata Iron & Steel Co. Ltd.*, Jamshedpur. The chief object of the work was to investigate the blending of non-coking coals or "feebly coking coals" with good coking coals in order to manufacture from the blend sound metallurgical coke. The work was undertaken in the interest of the conservation of the resources of the best coking coal.

### Quality of Coke

The chief desiderata in coke are purity, cohesion, size and uniform structure. These are governed mainly by the quality of coal charged into the oven but also by the bulk density of the charge, the temperature, rate of heating and duration of carbonization, the size and shape of the oven, the degree of grinding and thoroughness of mixing of the charge, its sulphur and moisture content, etc.

Regarding the special requirements of blast-furnace coke, uniformity is of great importance. Present variations in the physical and chemical properties of coke supplied to the furnaces in India are, broadly speaking, too great to permit of high efficiency in the furnace operations. This is partly true of the ash content of the coke which varies with the ash content of the coal supplied to the ovens. Uniformity requirements may indeed necessitate extensive washing of the coal supplies in the future in order to produce a practically uniform ash content.

Resistance to shattering and to abrasibility of the coke is also lowered by irregular

distribution of ash; and as strength, cohesion and size are to a degree inter-dependent and are of importance in furnace operations, it may be necessary to pay attention to the preparation of the coal before charging it into the ovens.

### Testing of Coal & Coke

The samples investigated were obtained from State Railways Coal Department and also through the agency of *Tata's Coal & Prospecting Departments*. Standard analytical tests were carried out including proximate analysis, determination of phosphorus and sulphur contents, caking index by Gray-Campredon method, swelling properties by Koppers and Nedelmann's apparatus and fusion range of ash. In addition, screen analysis, complete analysis of ash, determination of calorific value and softening point of coal were carried out in certain cases.

Laboratory blending tests were carried out as follows:

First, coals of possibly harmful swelling properties were blended with various other coking coals to study the effect of blending on the swelling characteristics.

Secondly, blends with normal coking coals, having a caking index of 15 (and above), were made with the following:

- (1) semi-coking coals of Gray-Campredon caking index 8 to 12;
- (2) poor coking coals (caking index below 8); and
- (3) non-coking coals, yielding non-coherent coke buttons.

Among the good coking coals employed were:

- (a) the usual mixture used in Tata's coke ovens, which is a blend in varying proportions of a group of low and medium volatile coking coals, almost entirely from the Jharia field;
- (b) low volatile, good coking coals from the Jharia coalfield, such as Sijua 12 seam or 12, 13, 14 and 15 mixed seams, etc.;
- (c) medium volatile, good coking coals such as Laikdih Deep, Laikdih seam, from the Raniganj field;
- (d) high volatile, good to fair coking coals such as Murulidih, Mohuda Bottom seam, as

well as some from the Raniganj field. A great variety of other coals from Jamadoba, Malkera, Choitodih, Digwadih, Bhatdee, Chanch and West Bokaro collieries were also used at the earlier stages.

Following the above laboratory work, semi-large-scale coking tests in specially designed electric (Swoboda) and/or gas-fired (Russell) pilot ovens were carried out to check the laboratory results.

Finally, full-scale tests in Tata's Simon-Carves coke ovens were conducted for confirmation of the results of the semi-large-scale work.

The blends for semi-large-scale and large-scale tests were submitted to proximate analysis and to tests for bulk density of the charge. The resulting coke was submitted to: (1) proximate analysis; (2) screen analysis; and (3) physical tests for shatter index, Breslau's abrasion, Haven's stability and porosity tests.

Under the head "Swelling Properties of Coal", the samples were classified after testing under three heads: (1) harmful; (2) doubtful; and (3) harmless. Typical curves obtained by the determinations carried out with the Koppers apparatus are given to characterize the three classes. According to Nedelmann's apparatus, dry coal (dried at 105°C.) having a bulk density of 51 lb. per cu. ft., a pressure of more than 14.22 lb./sq. in. developed during the tests is considered as "dangerous". For moist coal (10 per cent moisture) having a bulk density of 52.2 lb. per cu. ft., a pressure of more than 7.11 lb./sq. in. is considered the danger limit. If the pressure is below these limits, the coal is still regarded as "dangerous" if the shrinkage is low (less than 3 per cent).

*Pilot-plant Tests* — The coals were crushed in a William's patent pulverizer and crusher to a size conforming to the size and gradation obtained in actual coke oven practice (80 per cent through  $\frac{1}{8}$ "). The requisite proportions of crushed samples were subsequently mixed in an electrically driven mixer pan.

The pilot ovens employed for these tests were the Swoboda electric oven and the Russell gas-fired oven. The only difference in the oven practice is that in the case of the latter the coke mass is raked out from the chamber and quenched manually.

The coke obtained from the large-scale tests was subjected to screening to get the yield of commercial blast-furnace coke in different sizes as also those of nut coke, pearl coke and coke-breeze separately.

The following tests were conducted on the coke samples: (1) shatter index test on sizes over 2"; (2) Breslau's abrasion test (hardness) on sizes over 2"; (3) Haven's stability test on sizes between 2" and 3"; (4) porosity (including apparent specific gravity, real specific gravity and percentage porosity).

#### Large-scale Tests

Full oven tests were conducted in one of the coke ovens of the Tata's at Jamshedpur. The Simon-Carves Battery No. 3 of the "Underjet" compound regenerative type with twin-flue system of vertical heating was employed.

*Conclusions* — The general conclusions arrived at on the coking characteristics of various Indian coals are as follows:

1. *Caking Index (Agglutinating Value)* — Based on the experience gained from steel works practice it is considered that coals and blends with a minimum caking index value of 13 to 15 are suitable for producing satisfactory cokes.

2. *Moisture Content* — The tests on coals from Raniganj area, Central Provinces, Hyderabad and Assam showed that high natural moisture is associated with non-coking or poor coking properties.

3. *Swelling Properties* — There are hardly any coals in India which have to be classified in the category of harmful swelling coals. Furthermore, the addition of 5 to 10 per cent of other good coking coals of harmless swelling properties has been found to off-set the swelling tendencies of "possibly" dangerous coals as indicated by Koppers apparatus.

*Blending Possibilities* — Laboratory tests have demonstrated the blending possibilities of low grade coals to the following extent in admixture with good coking coals:

(i) 30 to 40 per cent for fair coking coals (caking index 13 to 14);

(ii) 20 to 30 per cent for semi-coking coals (caking index 8 to 12); and

(iii) 10 to 20 per cent for poor or non-coking coals (caking index below 8).

The above conclusions are valuable; they indicate a definite extension of lease of life for the coking coal reserves of the country. But there are also other valuable results such as the proof of the good coking properties of Begonia seam and of the blends with Dishergarh seam coals.

Neglecting the poor coking coals with a caking index below 8, it is observed that an average of 25 per cent of semi-coking coal

and 35 per cent of fair coking coal can be used in blends with good coking coal. The proportions are restricted because the Indian coals contain a large amount of "durain", which is basically a non-swelling and non-agglomerating constituent. Durains if interleaved with "brights" (Clarain) can give good coke. If a seam consists of alternating bands of layers of durain and "brights", it may give a sound metallurgical coke. This type of coal predominates in the Jharia coalfield.

The volatile matter content of the blend, it is suggested, should be kept sufficiently low in order that after carbonization the ash content in the resulting coke is low, the fixed carbon in the coke is sufficiently high, and excessive shrinkage is avoided.

In general, it has been found that higher the proportion of non-coking or feebly coking coals added to a good coking coal, the weaker is the coke produced.

#### Possibilities of Coals from Different Sources

*Jharia Coalfield Seams Below Seam 10* — The high ash content of these good to fair coking coals precludes their immediate utilization for coke manufacture unless they are used in blends with other good coking coals of sufficiently low ash. These high ash, good coking coals, therefore, along with several others of the semi-coking type, *should be washed* to reduce their ash content and thus render them suitable for coking industry.

*Raniganj Coals* — Dishergarh and Poniati seam coals, because of their better caking characteristics, seem to be usable in high proportions for the production of metallurgical coke. The behaviour of these coals will be examined further in semi-large-scale and full-oven tests.

The satisfactory utilization of the Begonia seam coal by itself, and of the Sanctoria seam to the extent of a 30 per cent blend with normal coke-oven coal mixture has been confirmed by full-oven tests.

The Begonia and Dishergarh coals are essentially bright coals, containing less durain than the coking coals of Jharia and are by themselves fairly good coking coals.

*Central Provinces Coals* — Of the coals so far tested, those from Hirdagarh, Kalichhaper, Damua and Rakhikol collieries seem to have moderate coking properties.

*Assam Coals* — Except for their excessive sulphur contents, Assam coals are often of good coking quality and have very little ash. High sulphur content, however, would seem to preclude their utilization for metallurgical purposes. A commercial process is required whereby the sulphur content is reduced appreciably, and the sulphur recovered if possible.

*Use of Coke-breeze in Blends* — The use of coke-breeze along with coking coals has not given promising results. Experimental work has revealed that its utilization to a limited extent of 2 per cent or so is possible only by fine grinding and by thorough mixing with the blend. Such a treatment in practice is both difficult and expensive.

#### Future Problems

The report points out that with regard to future work of the *Coal Blending & Coking Research Sub-Committee*, several full-scale verification tests have yet to be carried out on coals already tested on a smaller scale. The research programme envisaged for the future comprises:

(1) the study of the effect of variables such as oven width, temperature, and rate of heating and grain size of coals;

(2) assessing swelling and coking properties of coals in a more thorough and quantitative manner;

(3) standardization of analytical and testing procedure by carrying out tests with coal of known swelling and coking properties procured from abroad;

(4) investigation of special carbonization processes using blends of non-coking or poor coking coals in admixture with coal-tar or pitch.

# Famous Inventions: Bleaching Powder

**T**HE present year marks the one hundred and fiftieth anniversary of the first manufacture of bleaching powder by CHARLES TENNANT of Glasgow. This invention, patented in 1799, was the culmination of a number of discoveries dating back to 1773, in which year chlorine was discovered by Scheele. About a decade after Scheele's discovery, Berthollet first recorded the effect of the gas in bleaching the natural colour of linen. It was first applied to bleaching on a large scale by Berthollet himself in 1787. At this period the bleacher made his own chlorine and exposed the fabrics to the action of the gas in chambers or steeped them in its aqueous solution. The process was inconvenient and dangerous to the health of the workers. Considerable improvement was effected some years later by the introduction of "eau de Javelle" made by passing chlorine into sodium or potassium carbonate solution.

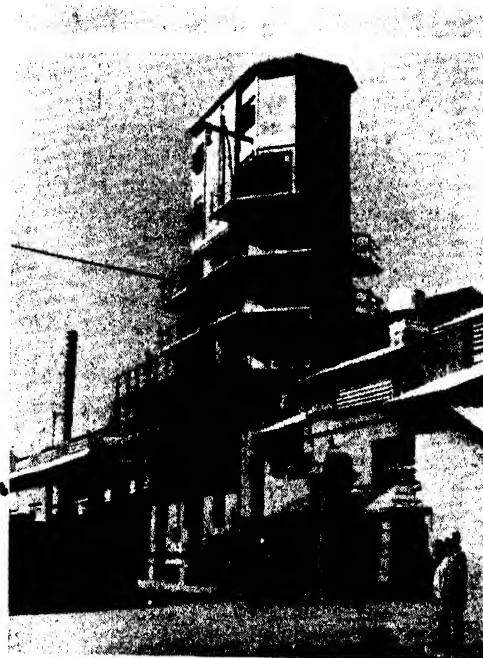
The next step was the preparation of a bleaching liquor by the passing of chlorine into a well-agitated mixture of lime and water. This process was patented by Charles Tennant in 1798. A number of Lancashire bleachers made use of the process without acknowledgement, and an action was brought against them by Tennant for infringement of his patent rights (Tennant vs. Slater). Tennant's suit was dismissed on the ground that the process had already been used by other persons prior to the date of Tennant's patent and that Tennant was not the *true* and *first* inventor of the process.

In 1799 Tennant followed up with his famous patent (No. 2312) for the manufacture of bleaching powder by the action of chlorine on slaked lime. He was the first to supply the bleacher with a reagent in the solid form, which contained up to one-third of its weight of available chlorine. Tennant's invention finally sealed the fate of the old bleaching process involving six to eight weeks of exposure of the material to be bleached to light, air and moisture. The open fields which were needed to spread the cloth for bleaching under the old process could thenceforward be utilized for other purposes.

Tennant's invention has given the world not merely an effective bleaching agent, but also a sanitary reagent of considerable importance and a substance with numerous other uses, for instance, in the manufacture of chloroform and the refining of petroleum oil. It is, in particular, an excellent substance for disinfecting water and even though it is often disliked and cursed for its chlorine-smell, it saves thousands of lives from infection by germs of typhoid and other deadly diseases.

Many attempts have been made to replace bleaching powder. This is possible where cheap electrical power is available for the manufacture of sodium hypochlorite from common salt. Another competitor of bleaching powder is liquid chlorine which can replace bleaching powder in many respects. The world output of the latter, however, still reaches many hundreds of thousands of tons yearly.

R. B. PAI



A MODERN BLEACHING POWDER PLANT AT THE METTUR  
CHEMICAL & INDUSTRIAL CORPORATION LTD.

# REVIEWS

**American Chemical Industry — The Merger Era**, by Williams Haynes (D. Van Nostrand Co. Inc., New York ; Macmillan & Co. Ltd., London ), 1947, Vol. IV, pp. xli+638. Price \$10.00 or 55s. net.

THE VOLUME UNDER REVIEW RECORDS THE growth of the American chemical industry during the period 1923-29. This period is sandwiched between two marked events in the economic history of the U.S.A. — the slump that had begun at the end of World War I and the financial crash of 1929. The Tariff Law was passed in 1922 and the effect of this upon general business was so electric that the period 1923-29 has been termed "The Booming Twenties".

It was in these Booming Twenties that the American chemical industry also underwent a complete metamorphosis. Helped by an enormous domestic market and abundant raw materials, the growth of the chemical industry was autocatalytic. The old rule-of-thumb methods were replaced by scientifically planned and tested ones. "Tanners, soap-boilers, paint-mixers, glassmakers — all became more chemically minded." "Urge to greater profits inspired numberless organized costly researches which brought to the market a multitude of cheaper and better products." It was a competitive era ; "competition was keen in the laboratories, the plants and the sales offices. It was the triple threat competition in which revised processes, new products and lower costs took turns in setting the pace of progress."

The intensity of competition throughout the chemical world created an atmosphere leading to amalgamations of smaller manufacturing units into larger ones. The pattern of these, however, varied from country to country. "Rigidly restricted by the anti-trust laws and liable at any time to be investigated by the Federal Trade Commission, American industries were unable to combine in the cartels and trusts typical of the movement in Europe." The consolidations called "mergers" by the author had, therefore, to be different. "Underlying the whole merger movement in the

chemical industry was a resilient chemical philosophy. Almost without exception the mergers, big and little, were based on the principle of diversification buttressed by chemical logic. Legal restrictions kept all producers of fine chemicals or alkalies or dyes from uniting into a single trust. But technological restrictions curbed a swing to the opposite extreme of gathering together a heterogeneous group of non-related chemical enterprises." Thus the mergers were a balance between diversification and unification. They were guided more by technical and economic advantages of large-scale operations, of the mass production of a single or inter-related chemicals and of the utilization of by-products rather than by the commercial advantages of handling a more complete line of chemicals.

With the above as the background and based on information and data contained in the contemporary technical and commercial journals, the author has written this, the 4th volume in the series *American Chemical Industry*, which makes a very delightful reading. It is not merely a collection of historical events and facts but a live running commentary on them ; "their meaning in the technical and commercial development of the industry, and their bearing upon the contemporary economic and political situation".

The distinguishing feature of this historical volume is that the narration of events is profusely interspersed with accounts of careers and achievements of several persons who contributed largely to the chemico-industrial development — chemists in laboratories and in plants, chemical engineers, economists, executives of various companies and committees, salesmen, Government officials and even politicians. The reader is thus constantly made aware of the enormous efforts, talent and co-operation that lay behind the successful promotion of numerous companies, manufacture of the various products and their sale. The volume is indeed a tribute to the scientific, technical and commercial genius of the Americans.

The main text of the volume is divided into 5 parts which are spread over 26 chapters

in all. The 1st part deals with the then general background of the chemical industry and shows how the booming automobile, motion picture and radio industries created new and vast chemical demands. It gives very vividly the story of big mergers which brought into being *du Pont*, *Allied Chemical & Dye Corporation*, *Union Carbide & Carbon Corporation*, the *Atlas Powder Company*, the *Hercules Powder Company*, *Mon-santo Chemical Works*, the *American Cyanamid Company*— names that are now household words. In fact, some 500 mergers were completed among chemical companies in this period.

The 2nd and the 3rd parts deal with the growth of inorganic and organic chemical industries respectively. Part 4 is concerned with medicinal and other fine chemicals while the consuming industries such as fertilizers, plastics, paints, rayon and petroleum are considered in the 5th part.

To a country like ours which is embarking on a rapid industrialization, this volume is a great pointer. It will enable us to weigh our progress in fields already established like the heavy chemicals, wood chemicals, pharmaceuticals, etc., and make us wise to the complexities of the industries to be established like those of fertilizers, rayon, plastics, dyes, etc. The misfortunes of alcohol and the plight of its legitimate industrial users under the American prohibition regime as related in a separate chapter are worth serious consideration in the context of this country's prohibition programme. Thanks to the over-zealous prohibition officials, use of alcohol as a solvent and a chemical was ignored and even the use of denatured alcohol was restricted. The situation arising out of this must have indeed been unbearable since alcohol then came to be defined not as a beverage, a solvent or a chemical, but as "a distillation of politics denatured with the unadulterated essence of prejudice".

The usefulness of the volume has been further increased by several other features such as a chemical chronology, 1923-29, profuse foot notes giving critical views of several authorities on points at issue, and 49 appendices most of which give in tabular form statistical data on production, exports and imports of important chemicals and the rest of which are special notes contributed by others on certain topics. The volume is illustrated with portraits of personalities that made the chemical era of 1923-29.

The book lacks nothing in its thoroughness, in its printing and in its get-up. Anyone interested in chemical industry, be he a chemist or non-chemist, is assured of a pleasant and fruitful reading.

J. G. KANE

### **Spectroscopy and Combustion Theory,**

by A. G. Gaydon: (Chapman & Hall Ltd., London), second edition, 1948, pp. xii+242. Price 25s. net.

THE APPLICATION OF SPECTROSCOPIC THEORY and technique to the problems of fuel combustion has been comparatively a recent growth. Dr. Gaydon, an active worker in this field, published in 1942 a book incorporating information then available on this very vital subject. The advances in this field of knowledge have been so quick that a new edition was thought imperative, and Dr. Gaydon, in this second edition, has collected as much material as was available up to the time of his writing.

The book devotes 2 chapters to the elucidation of the fundamentals of the theory of atomic and molecular spectra which form the basis of the subject. The account may appear sketchy to a general reader, but then it could not be otherwise in a volume devoted chiefly to the applications of spectroscopy to the combustion theory.

The chief distinction, as stated by Dr. Gaydon in the introduction, between combustion and ordinary chemical reaction is the appearance of a flame, i.e. the emission of light. Thus, while the chemical study provides knowledge of certain broad stages of combustion mechanism through the stable products, the spectral study enables intermediate processes to be observed and interpreted in terms of the physical radicals  $C_2$ ,  $CH$ ,  $OH$ ,  $CHO$ , etc., by their characteristic radiation. Dr. Gaydon deals with such spectral products of different flames, and discusses their appearance in terms of the flame condition. Experimental study in this field has developed in a somewhat *ad hoc* manner, and the correlation of data to get at the central idea of the combustion mechanism is the author's aim in this treatise.

Dr. Gaydon begins with the spectral radiations of elementary flames such as those of burning hydrogen, of hydrocarbons like ethylene and methane, cool flames and carbon monoxide flame, and then goes on

to explosion spectra in the internal combustion engines. The most important spectrum, from the point of view of combustion theory, is that of hydrocarbon flames, the bands of which Vaidya has tentatively shown to be due to CHO. Dr. Gaydon discusses all evidences in favour and against this emitter and shows that further crucial tests are necessary to confirm it. According to the latest information which the reviewer has in his possession (through private communication with Dr. Vaidya), the problem is nearing the expected solution consequent on the identification of the spectrum of deuterio-acetylene. Many other relevant aspects, e.g. the continuous spectra, absorption spectra, infra-red spectra, dissociation energy, flame temperatures, after-burning and life-times of excited molecules, energy equilibrium, inorganic flames, etc., have been dealt with and their specific bearing on combustion discussed.

There are, however, a few points on which some criticism may be offered. To one intent on grasping the ideas relating to combustion, the plan of division and distribution of the subject-matter among chapters is not quite obvious. The logical approach would have been first to treat the kinds of flames and the techniques of excitation, viz. low pressure, atomic, cool flames, etc., followed by a discussion on the bearing of each type of spectrum—emission, absorption, continuum, infra-red—on the combustion theory. Each of the flames would then form a complete topic in itself, and overlapping and repetition in different chapters would be avoided. Such an arrangement is likely to give a more coherent picture of the combustion mechanism and enable the discussion of the hydroxylation and peroxidation theories, now discussed under hydrocarbon flames, continuous spectrum and cool flames at a single concluding stage. Though attempts have been made to discuss the bearing of effective temperatures on the equipartition of energy in a flame in chapters XIV and XVI, some of the important quantitative results available in literature have not been referred to as evidence supporting the points brought out. In discussing the effect of transition probability and of collisions in determining the strength of emission spectra, Dr. Gaydon observes that a remarkable change in the relative intensities of the nitrogen 1st and 2nd positive systems in air is due to the 20 per cent oxygen. While this may be true, the

available experimental data does not positively lead to that conclusion, as traces of rare gases, also present in air, or their admixture with nitrogen have been shown by Johnson, and lately by Tawde and Patankar to cause a redistribution of intensity. Again on page 194, the rôle of relative intensities of bands in a system has been mentioned only briefly when the same could have been subjected to a more detailed examination.

The few omissions or shortcomings noted above are not such as to mar the value of the publication which is a new approach to the subject of combustion. Dr. Gaydon is to be congratulated on revising his first edition of the book by rewriting some parts and adding to it a substantial 50 pages or so of important research matter gathered during the intervening period of 5 years. The appendices at the end are particularly aimed at assisting an active spectroscopist. The book, as a whole, will appeal both to the general physicist and chemist, and is a significant contribution to the literature on combustion.

N. R. T.

**Non-Ferrous Castings**, by R. F. Hudson (Chapman & Hall Ltd., London), 1948, pp. ix+282+xxiii. Price 22s. net.

IN HIS PREFACE THE AUTHOR STATES THAT the main purpose of this book is to bring the scientific view-point of non-ferrous metallurgical operations to the practical man in a language he understands and to give a simple outline of the modern methods employed.

For a very long time the process involved in casting was considered to be highly secretive, but in recent years, with the advent of *British Standards Specifications*, the full particulars of the constituent metals are available. But, however, some knowledge is required of the mixing of metals and also on the correct pouring temperature. This information cannot be found in any book and one has to acquire it only through constant practice. If technicians connected with foundry could publish their experiences acquired through long years, it would contribute to the progress of this branch of engineering and also would be to the advantage of the young men entering this industry.

The entire matter is presented in 9 chapters commencing with a short description



of the main in-coming materials such as metals and fuels which the non-ferrous foundries are likely to handle. Moulding, melting and gating practice for most of the standard and nickel base alloys are very exhaustively treated and quite a good deal of space is devoted on this aspect by the author. He has also made an endeavour to outline a few recommended melting procedures which are likely to yield good results.

The book ends with two appendices, one showing the photographs of the defects most commonly found in the non-ferrous foundry practice with likely causes and cures. The second appendix shows some photo-micrographs of the more common non-ferrous alloys indicating the various constituents and their effect on physical and working properties of sand, chill and centrifugal casting.

In all aspects, this treatise can be said to have covered all modern methods which were employed in foundries during the war and there is hardly any topic which has escaped the attention of the author.

This book can be used in all foundries as a reference book and it will be a valuable addition to the desk of foundry technologists as well as metallurgists.

V. CADAMBE

### **Solid and Laminated Wood Bending,**

by W. C. Stevens & N. Turner (His Majesty's Stationery Office, London), 1948, pp. viii+71. Price 5s.

IN SPITE OF WOOD BENDING BEING AN ancient art dating back to the period when man first learnt to make baskets and boats from wood, there has not been much work done in this field till recently. Owing to timber shortage, the problem of wood bending was taken up by the *Forest Product Research Laboratory* with which the authors of the book under review were associated for nearly 15 years.

In this treatise the authors have presented a very useful material scientifically elaborating the various processes involved in wood bending. To the best knowledge of the reviewer, this book seems to be the first of its kind in the field, and, therefore, should prove to be a valuable addition to the desk of all those connected with wood in some form or the other.

There are many practical suggestions supported by adequate theoretical investiga-

tions for the economic use of wood for various purposes. The subject is treated systematically, the first half of the book dealing with solid wood bending and the latter half with laminated wood bending. It is remarkable to find that the authors have made available a large number of plates in this small treatise to illustrate the methods employed for accomplishing the many types of wood bending described in the body of the book.

The chapters on laminated bending deserve special emphasis in that many new and tested processes are described which should be very useful to all those engaged in the manufacture of furniture and other allied industries where wood forms the basic material.

V. CADAMBE

### **Standards For Testing Soils ( Tentative )** ( Published by the Central Board of Irrigation, Simla ), 1948, pp. 195+iv.

ALTHOUGH SOIL HAS BEEN USED AS A CONSTRUCTIONAL material since prehistoric times, the study of the engineering properties of this material had lagged behind the general progress in engineering science. The study of soil mechanics and the related physico-chemical properties is comparatively of recent years, although soil in its relationship to crop growth has been studied for over a century.

All those engaged on research in soils as an engineering material would welcome the book *Standards For Testing Soils ( Tentative )* published under the auspices of the *Central Board of Irrigation*, Simla. It is only fitting that the Board with its official status and widespread activities in the field of irrigation research should have taken a lead in bringing forth such a volume "to enable full benefits to be derived from comparison of results and to ensure uniformity of practice in research and routine work".

The publication is the result of efforts of a permanent Committee of the Board appointed in 1945 which included representatives of the Railway Board, Roads Organization and Army Engineering Corps. It would have been better if the Committee had also included a soil chemist who could have helped in the elucidation of the other aspects of soil science. The book gives methods for testing soils as required in engineering practice only. In a similar booklet issued by the States Rivers & Water Supply

Commission, Australia, the title of the book is *Standards for Testing Soils as practised in a Soil Mechanics Laboratory*. Some such title would have been appropriate.

The book has been divided up into 6 chapters. The 1st chapter is the introduction, while the 2nd enumerates the various tests necessary for each of the following :

1. Roads and Air-field Sub-grade.
2. Low Cost Roads.
3. Earth Dams.
4. Embankments.
5. Foundations for Bridges, Buildings and Dams.

Separate tests for routine and research work have been recommended for the above.

The 3rd chapter deals with the surveying methods and methods for soil sampling. It would have been better if many of the details such as the survey forms, sheets and other information had been included as an appendix at the end of the book. The next chapter describes the preparation of samples for different tests while the last two chapters cover up all the 24 tests — physico-chemical and mechanical. Leaving aside the 7 chemical tests (which have been disposed off in 7 pages), the other tests have been detailed in 150 pages out of which about one-third space has been devoted to mechanical analysis alone. It should have been easily possible to insert some of the details, such as Tables V to XIII, in the appendix.

In addition to the inadequate attention paid to the chemical tests, their description is rather incomplete and is not properly put. For example, the method described on page 36 under "hygroscopic moisture" is actually the "oven dry moisture". These two kinds of moisture are quite different from each other and the laboratory worker is likely to be confused. Similarly the colour of a soil sample has to be examined in the dry as well as in the wet condition. It would have been beneficial in the interest of the workers if the difference between *soil texture* and *soil structure* had been clearly indicated.

It is doubtful whether recommendations made in the book as regards the total quantity of soil required for different tests is correct. In the reviewer's opinion the quantity is only 50 per cent of that actually required. Approximately 2 lb. of sieved soil must be made available for complete analysis of a soil.

In the determination of total soluble salts the book prescribes a soil water ratio of 1:10. However, in all standard works 1:5 soil water ratio is used and our work also justifies this. The quantity of water extracts to be evaporated for the determination of soluble salts must not be less than 100-150 c.c., so that there would be less chances of errors in the final calculation. It is doubtful whether the rhodozonc acid method of estimating sulphates should be used, as the method is not adequately standardized.

The estimation of soil organic matter (organic carbon) is important in all soil work. But a single method cannot be recommended safely for all types of soils specially in the case of saline and calcareous soils. Therefore, it would have been proper if some standard methods had been recommended with their limits of applicability indicated. Walkley and Black's rapid titration method is a quite useful method for organic carbon determination. The cation exchange capacity is always expressed in milli-equivalents per 100 gm. of oven dry soil. However, in the method described on page 190 (which appears to be Puri's ammonium carbonate method) this point seems to be omitted inadvertently.

In general, it can be said that in all the above tests, specially the chemical tests, it would have been a wiser policy to give a short review of the different methods of analysis with their scope and then suggest methods for particular kind of soil type in India.

The *Central Board of Irrigation* has done a great service to soil analysts in India by bringing out this useful publication. They also deserve credit for making available the diagrams, specifications and sketches of many useful appliances and equipment so very necessary for the various tests and which could easily be constructed in any good workshop.

It is suggested that when these tentative methods are revised, it would be better to split this book into three parts, viz. :

1. Road, Low Cost Roads and Air-field Sub-grades.
2. Earth Dams and Embankments.
3. Foundation Problems.

It would then be possible for workers engaged in a particular kind of problem to choose those tests suitable for their work.

# NOTES & NEWS

## Isolation of Vitamin A<sub>1</sub>

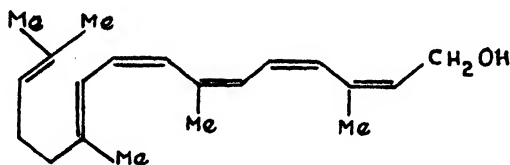
SINCE THE DISCOVERY IN 1937 OF vitamin A<sub>2</sub>, a compound related to vitamin A and found chiefly in the livers of certain species of fresh-water fish, there has been much disagreement among investigators with respect to its structure and biological activity. The reported isolation of pure (though non-crystalline) vitamin A<sub>2</sub> alcohol through a crystalline ester established criteria by which the activity of vitamin A<sub>2</sub> can be measured (*Science*, 1948, **108**, 417).

Fish livers exhibiting the characteristic brownish-orange fluorescence of vitamin A<sub>2</sub> were ground and extracted with ethyl ether to obtain a low-potency oil (potency index, 4). The concentration of vitamin A<sub>2</sub> is carried out by twice distilling the low-potency oil in a centrifugal molecular still to obtain a concentrate with a potency index of 65. Separation of the triglycerides by saponification increased the potency index to 182 and the sterols crystallized out in acetone at  $-30^{\circ}\text{C}$ . had a potency index of 260. The residue on chromatographing on sodium-aluminium silicate, distilling chromatographed concentrate in a high-vacuum pot still, destero-lating again in ethyl formate at  $-30^{\circ}\text{C}$ ., chromatographing on magnesium oxide and thrice on zinc carbonate, a concentrate with a potency index of 1,350 was obtained. Further concentration could not be effected. The concentrate was esterified with phenylazobenzoyl chloride by allowing it to stand for 4 hr. at room temperature in methylene chloride containing pyridine.

The vitamin A<sub>2</sub> phenylazobenzoate thus obtained was chromatographed twice on zinc carbonate and eluted with petrol ether (Skellysolve "F") and allowed to stand in the solvent at  $-30^{\circ}\text{C}$ . for 3 days when hemispherical orange rosettes were obtained (m.p.  $76-77^{\circ}\text{C}$ .). The compound had an ultraviolet adsorption maximum at  $341\text{ m}\mu$  of 1,190. Careful saponification of the crystals under nitrogen gave theoretical yields of vitamin A<sub>2</sub> alcohol which gave a single homogeneous adsorption band on a zinc-carbonate column with two tiny

adsorbed bands. The eluted material from the homogeneous column was a viscous, orange-yellow oil which showed 2 adsorption maxima in the ultraviolet at  $351\text{ m}\mu$  and a subsidiary peak at  $287\text{ m}\mu$ . A chloroform solution of antimony trichloride product of vitamin A<sub>2</sub> gave a blue colour with a single adsorption maximum at  $693\text{ m}\mu$ . The chromatographically concentrated anhydro-vitamin A<sub>2</sub> (obtained by treating vitamin A<sub>2</sub> alcohol fractions with N/30 alcoholic hydrochloric acid) readily crystallizes at  $-30^{\circ}\text{C}$ . from a 20 per cent solution in light petroleum (Skellysolve "F"); the crystals melting at  $89.5^{\circ}\text{C}$ . The anhydro-vitamin A<sub>2</sub> is easily separated from its analogous anhydro-vitamin A, by adsorption on zinc carbonate whereas the vitamin A and A<sub>2</sub> alcohols are difficult to separate.

The following structure has been suggested as the most probable:



Biological tests indicate that vitamin A<sub>2</sub> alcohol has a potency approximately 1,300,000 U.S.P. units/gm., or about 40 per cent the activity of crystalline vitamin A.

## Mutations by Uranium Nitrate

THE ADDITION OF URANIUM nitrate  $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , to potato-dextrose agar at the rate of 0.5 to 1.0 gm./litre has been found to stimulate mutation in the cultivated mushroom, *Agaricus campestris*, and both mutation and an unusual type of dissociation in the ordinary corn smut fungus, *Ustilago zeae* (*Science*, 1948, **108**, 554).

Experiments on corn smut fungus show that uranium nitrate induces reduction division or some other type of nuclear change resulting in the dissociation of cultural, and pathogenic characters of certain monosporoidal haploid lines. There is evidence

also that the number of mutants produced in liquid media containing uranium nitrate may be even greater than on agar.

Uranium nitrate and other uranium salts have induced mutants in *A. campestris* whose growth on artificial media is from 5 to 7 times that of the original lines, as determined by dry weight of mycelial mat. Moreover, spawn of some mutant lines has produced mushrooms earlier than the checks and the colour was white instead of brownish.

The agar containing uranium nitrate is mildly radioactive. It is suggested that the addition of uranium nitrate or other salts, to nutrient media may be a simple and useful means of inducing desirable mutations in micro-organisms.

## Blanc Fixe from Barytes

NEW PROCESSES FOR THE PRODUCTION of blanc fixe and barium carbonate from barytes and of strontium carbonate from celestite are described (C.T.J., 1948, **123**, 563). The methods are based on the fact that barium sulphate is soluble in fused sodium chloride and that on quenching the melt with water the barium sulphate is

reprecipitated. In the new process, the discolouration of the finished product is overcome by eliminating the iron oxide impurity.

The furnace used in pilot plant tests is made of a 10' long welded steel shell 0.5" thick and lined 9" thick with an all-clay firebrick. The poorest grade of firebrick was found to be the best. Fuel oil burners are placed above the melt at such a height that gases do not impinge upon the melt. The furnace atmosphere must be oxidizing. The salt-barytes mixture is blown into the furnace by a compressed air jet at right angles to the flame jets.

The impurities, save for a trace of iron oxide, settle to the bottom of the melt and are removed periodically by tipping the furnace forward and raking out the residue. A baffle prevents the residue from moving forward with the melt. Another baffle prevents floating impurities from contaminating the product.

The decolorizing material consists of a mixture of 99.9 per cent sodium chloride and 0.1 per cent

commercial phosphate fertilizer, blown over the surface of the melt just before it leaves the furnace. The melt is quenched in an air-agitated tank, washed, filter-pressed and tray-dried.

It is necessary that barytes and salt are dried before charging them into the furnace to prevent formation of hydrogen chloride. The mixture is ground together to 16 mesh.

The barium sulphate produced is a brilliant blue-white material of an apparent gravity about one-half that of precipitated product. It contains no free sulphur or sulphides and is suitable for X-ray medical use.

In the production of barium carbonate from barytes, also developed to the pilot plant stage, the crude barytes is fused with sodium chloride as above and after the insoluble impurities are separated by settling and decantation, sodium carbonate is added to the melt. Under conditions of slow cooling the barium sulphate is converted to carbonate which is separated by leaching out other salts. A similar procedure is adopted for converting celestine into strontium carbonate.

### Production of Thin Metal Films

EXTREMELY THIN METAL FILMS were previously obtained by evaporating a small amount of a metal, e.g. beryllium or aluminium, on the surface of a film of cellulose nitrate and subsequently dissolving away the cellulose-acetate film by amyl acetate or other appropriate solvent. Such films are especially useful as supporting membranes for electron microscopical studies. Though, as a rule, these films do not exhibit any visible structure, a faint structure may make its appearance. This structure-effect of the films has been eliminated by using acrylates instead of cellulose nitrate, collodion, etc., as the basic film. The solvent employed is ethyl acetate (*Nature*, 1948, 162, 892).

The metal films so obtained are almost invisible in the electron microscope when made 20Å in thickness and the photographic contrast effect of the object-particles is increased compared with that obtained with other supporting membranes.

Another method, which is simpler because no dissolving process for the basic film is used, consists in the evaporation, in

vacuum, of a small amount of beryllium or aluminium on to a liquid surface. Glycerol or any other liquid having a high-boiling point and low-vapour pressure at room temperature is suitable. A piece of glass, about 3 cm.  $\times$  3 cm., is covered with a thin layer of glycerol ( $\frac{1}{8}$  mm. or less in thickness) and put into the vacuum chamber. After a while, when water which may have been absorbed in the glycerol has evaporated, a small amount of metal is evaporated. The glass slope is next put in a container into which distilled water is introduced. The glycerol is dissolved and the film immediately floats off; it lies on the water surface, holding together well. The film is washed by using a container in which the water can be changed without disturbing the film. The film is transferred to a plane grid of copper, platinum or other metal, the holes measuring about 0.1 mm. in diameter, and is allowed to dry. In this way it is easy to get pieces 3 cm.  $\times$  3 cm. or more, and whenever a specimen is needed, a little piece can be cut off from this ready-made film.

It is possible to make membranes of beryllium, aluminium, etc., with a material thickness of only 10-20Å, lying on grids where 80 per cent of the area is covered with only the thin metal film. One of the greatest advantages of this method is the possibility of laying a metal film directly on an organic substance without using amyl acetate or other liquids which affect organic substances.

### Benzoic Acid Manufacture in Germany

A SURVEY OF THE PLANT AND methods used in the manufacture of benzoic acid in Germany has been made in the BIOS Final Report No. 1786 (C.T.J., 1948, 123, 595). The production of benzoic acid and benzaldehyde was confined to 2 plants of I.G. at Ludwigshafen and Uerdingen.

Phthalic acid was the raw material employed at Ludwigshafen. One phthalic anhydride unit incorporated a catalytic decarboxylation converter which could be by-passed as and when benzoic acid was not required. The classical method for the production of benzaldehyde via side-chain chlorination of toluene was in operation at Uerdingen and gave benzoic acid as a by-product. Benzoic acid was also manufactured at this plant by the bichromate oxida-

tion of toluene in the liquid phase. Because of the high cost, bichromate oxidation is likely to be superseded by the air-oxidation process, but was still employed for the production *para* and *ortho*-chloro benzoic acids. The greater portion of the benzoic acid produced was neutralized and sold as sodium salt.

Benzoic acid is obtained in 80 per cent theoretical yield by the liquid-phase air oxidation of toluene in the presence of a soluble cobalt salt. Air is bubbled through toluene containing 1 part per 1,000 of cobalt oleate, the toluene temperature being maintained at 140°C. and the whole operation being effected at 2 atm.; water, formic acid and carbon dioxide are removed continuously while the benzoic-acid content of the toluene builds up to 50 per cent. At this stage the benzoic acid is washed out with sodium carbonate solution, the resulting sodium benzoate solution being purified by activated carbon and then drum-dried. Benzylbenzoate is formed as a by-product and is recovered as a residue from the toluene recovery for subsequent hydrolysis to sodium benzoate and benzyl alcohol.

Sodium benzoate is produced continuously in 92 per cent theoretical yield by the pressure oxidation of toluene, using sodium bichromate as the oxidizing agent. A 33 per cent solution of benzoic acid in toluene and a 34 per cent sodium bichromate solution are intimately mixed as the solutions are pumped through a series of autoclaves in which the temperatures are held at 300-310°C. and which are under pressure of 200 atm. Residual bichromate is reduced by formaldehyde. After cooling, the unreacted toluene is removed by distillation, chromium hydroxide filtered off and the sodium benzoate solution is carbon-treated prior to passing to drum-dryer. Benzoic acid is obtained as a by-product in the benzaldehyde manufacture along with benzotrichloride. Chlorine is bubbled through toluene which is initially warmed to 100°C. and permitted to reach a temperature of 145°C. at the end of the reaction which normally takes up to 110 hr. The mixture then consisting of 65 per cent benzal chloride and 35 per cent benzotrichloride is run into 76 per cent sulphuric acid at 60°C. After dilution, the benzoic acid oily layer is removed by sodium carbonate and recovered from the aqueous resi-

due by the addition of hydrochloric acid.

In the Ludwigshafen process employing phthalic anhydride (obtained from naphthalene) the operation is conducted in the vapour phase by passing the gases from the phthalic anhydride converter (sufficient water vapour present) through a catalyst bed consisting of zinc oxide and aluminium oxide on pumice. The crude benzoic acid in water suspension is treated with sodium bisulphite in a Nutsche filter, centrifuged and washed free of electrolytes and sodium phthalate. It is finally sublimed for further purification or used as such for the manufacture of sodium benzoate.

### Rubber Chemicals in Germany

A RECENT BIOS REPORT DEALS with the German synthetic rubbers, "Bunas" and "Perbunas", and includes interesting sections on compounding ingredients (accelerators, anti-oxidants, plasticizers, softeners and pigments) (*C.T.J.*, 1948, 123, 616).

Among the reinforcing and general fillers special prominence is given to finely divided aluminium hydroxide, carbon black and a special form of silicon dioxide. The aluminium compound "Tonerdegel" or "Teg" is obtained by the double decomposition of solutions of aluminium sulphate and sodium carbonate, filtering the precipitate, drying and crushing. There appears to be a residuum of  $\text{SO}_2$  groups firmly bound in the hydroxide and largely responsible for the effect of the filler in rubber mixes. The gel is claimed to have given good results in shoe soles, jar rings, bottle discs and dipped gloves. Proportions up to 40 per cent on the weight of the mixing have been employed.

The "white carbon" is a finely divided amorphous silica produced by chlorinating 90 per cent ferrosilicon, burning the silicon tetrachloride in a mixture of air and hydrogen and collecting the silica smoke in a revolving drum. Particle sizes range from 0.1 to 1.0  $\mu$ , the finest grade being smaller than the finest carbon blacks. On the utility of this material the opinion has been unanimous.

Zinc oxide, one of the inorganic accelerators produced by the I.G., is made by precipitating zinc chloride solution with sodium carbonate and calcining the zinc carbonate at 450°C. The resulting zinc oxide is claimed to have

a smaller particle size than any other zinc oxide produced so far. Among the organic accelerators the important ones are mercaptobenzothiazole (Vulcacit M) and dibenzthiazyl disulphide (Vulcacit DM). Phenyl- $\beta$ -naphthylamine and aldol-*l*-naphthylamine were widely employed as organic anti-oxidants.

Oxycresylcamphene was found to be the best stabilizer for raw "Buna" rubber.

A wide range of plasticizers were investigated. One of the important materials is "Koresin" of I.G., a condensation product of acetylene and *p*-tertiary butyl phenol. Numerous esters of phthalic acid were also used as plasticizers under the trade name "Palatinols". Thio- $\beta$ -naphthol ("Renacit" of I.G.) was one of the many products introduced as softeners and reclaiming agents.

Considerable work is reported on the production of oil factices (rubber substitutes). One particular range of products were made by vulcanization by the Peachey process. As rape seed oil was in short supply the German factice industry employed whale oil, seal oil, sardine oil and linseed oil.

The report contains useful manufacturing details for synthetic iron-oxide pigments, zinc sulphide, etc., all types of goods made from rubber or rubber latex and analysis and testing of raw materials and finished products.

### Freeze Concentration

A PROCESS, WHICH MAY EXERT A revolutionary effect on the concentration of solutions of heat-sensitive materials, is now approaching the commercial stage at the *Commonwealth Engineering Co., Ohio* (*Chem. Eng.*, 1948, 55, 119). The process makes use of new principles in freezing to remove a large part of the water from the original solution, with only negligible loss of valuable dissolved material. A novel application of the method which may develop is the concentration of extremely corrosive solutions.

What is new in the process—called Cunningham Process after its inventor—is the employment of controlled ice-crystal growth in such a way as to effect a clean separation of ice and concentrate, with negligible loss of valuable material in the ice. In the concentration of fruit juice, for example, the ice carries with it only about 1 per cent of sugar initially present, in spite of the fact

that nearly 90 per cent of the original water is removed as ice.

Contrary to the usual freeze-concentration procedure, the new process grows the ice crystals in several stages and in a counter-current relation to the flow of solution so that the largest crystals are in contact with the most dilute and least viscous solution and a simple centrifuging is all that is needed to effect nearly complete separation. The only limitation of the process, apart from its economic aspect, is that concentration cannot be carried quite as far as the eutectic point.

The operational details are: the pre-cooled solution (work has been done with orange juice) enters the first of a cascade of 4 semi-cylindrical metal troughs and super-cooled to  $-10^\circ\text{C}$ . in the second trough. The troughs are provided with a slowly moving screen each of which separates the suspended ice crystals from the solution. Ice forms immediately and concentration increases as the viscous solution passes on to the third trough and then to the last. When the solution reaches the last trough the total percentage of solids is about 68 and the temperature is about  $-30^\circ\text{C}$ . At this concentration the product may be frozen or kept refrigerated for shipment. This is unnecessary in the case of fruit juices since the sugar content is high enough to be self-preserving.

### Wood-pulping with Magnesia

THE FIRST PULP MILL EMPLOYING magnesia for pulping wood will go into production shortly at the *Weyerhaeuser Timber Co., Longview, Washington*. The usual waste liquor disposal problem is eliminated in the cyclic magnesia-base sulphite process as the chemicals employed are recovered and reused. The advent of a cyclic recovery system in the sulphite pulp process, comparable in results with the cyclic recovery system used in the soda and sulphate processes, is expected to have very important implications for the future of wood pulp industry and is regarded as one of the outstanding advances in paper industry. (*Chem. Age*, 1948, 59, 724).

The waste liquor recovery system is a further step in the complete use of forest products. Complementing the recovery of chemicals, in the form of magnesium oxide and sulphur dioxide, is the production of heat and electrical energy which will supply

a large part of the power necessary to operate the mill.

The waste liquor (12 to 14 per cent solids) before concentration is neutralized with recovered magnesium oxide to prevent loss of any sulphur dioxide during evaporation. The concentrated waste liquor is burned in a recovery furnace under self-sustaining combustion conditions for steam generation and the highly reactive ash in the flue gases are collected in cyclonic separators. The cooled flue gases containing sulphur dioxide are combined with magnesia slurry to complete the cycle.

Pulp of a better quality than calcium-base pulp is obtained with the magnesia process because the greater solubility of magnesium compounds facilitate the pulp-washing operations resulting in fewer impurities in the finished pulp. Much of the slab wood and trimmings used to feed the boilers is now used up to produce pulp.

The process is available for paper industry by licensing under patents by the *Babcock & Wilcox Company*.

### Mechanical Mica-splitting Machine

FOLLOWING A STUDY, BEGUN during the war, of methods of splitting mica, the *U.S. National Bureau of Standards* have developed a mechanical mica splitter which facilitates the production of thin films. The machine not only promises to speed up the process considerably but will greatly reduce the period required to train skilled splitters.

Due to common mica imperfections such as unequal spacing of cleavage planes, no precise control of film thickness has been obtained. The quality of the machine split films, however, compares favourably with that of hand-split films. In tests at the *Bureau*, the rate of splitting by an untrained operator was 60 films a minute, a rate that could be increased with skill in operation. The average rate of hand-splitting is between 15-20 films per minute.

Like a hand splitter the machine starts thin sheets by introducing a sharp point into the edge of the mica block. A master valve at the centre of the turntable connects the chuck to the vacuum line to hold the block in position while it is being split; on travelling forward, a carriage moves the chuck a short distance, causing the splitter gauge to raise a film of mica. As the carriage returns to its starting position, 2 stripper

blades pass under the raised film to separate it from the block. The free end of the conveyor draws the loosened film to the belt which carries it to a receptacle. The use of an electromagnet to obtain a rapid vibration of the stripper is contemplated to increase the effectiveness of splitting (*Chem. Age*, 1948, 59, 731).

### Prevention of Potato Dry Rot

A HIGHLY EFFICIENT PREPARATION for the prevention of dry rot of potato tubers caused by the fungus *Fusarium coeruleum* has been claimed at the *Crawley Research Station of the Bayer Products Ltd.* This preparation marketed under the trade name of "Fusarex" is based on 2, 3, 5, 6 tetrachloronitrobenzene. "Fusarex" is most effective for the control of dry rot when applied to the tubers immediately on lifting, followed by a period of 4 to 6 weeks' storage. It has been found in trials that the preparation does not need to cover each potato completely so long as an even distribution through the mass of tubers is obtained. Thus a simple manual application can be employed for treating the potatoes. It has also been found that "Fusarex" treatment and storage conditions can delay the development of sprouts by the tubers. Pharmacological tests have shown that tetrachloronitrobenzene is non-poisonous under the conditions of use and has very low toxicity (*Nature*, 1948, 162, 843).

### New Uses for Porous Metal

SOME OF THE CURRENT APPLICATIONS of powder metallurgy are reviewed in a recent number of *Chemical Age* (1948, 59, 756).

Among the newer developments, the two principal ones are the production of pre-cast powders and the hot pressing of powders. The latter procedure offers the advantage of working at lower pressures.

Light metal alloys, e.g. aluminium alloys, are obtained by a combination of hot and cold pressing. The method eliminates the deleterious effects of temperatures above 200°C. The specific weight of the new alloys from powders should not exceed 5 gm./cc., preferably 3 gm., with a tensile strength exceeding 30 kg./sq. mm. even in the annealed state. These alloys are prepared by compressing and sintering aluminium powder, pure or alloyed, of such fineness that at least 50 per cent

has particle fineness <2 $\mu$ . The alloy constituents may be magnesium, copper, and/or silicon. The order of cold or hot pressing may be interchanged followed by sintering at the appropriate stage. Cold pressing is done at 2 tons/sq. cm. and hot pressing is done above 400°C.; preferably 550°-600°C.

Hot working, such as extrusion at about 450°C., is also said to be beneficial. Control of oxide content is important, for some of the unexpected properties obtained are due to oxide films. The products are particularly suitable for use at high temperatures, for internal combustion engine pistons and rings, turbine blades, etc.

Another new development is in the filtration and allied fields. New alloys of copper-nickel, copper-nickel-tin, etc., have been developed to give an immense range of porosity of the material which is also dependent on the thickness of the material. The material can be bonded with steel, copper, nickel and their alloys and made integral with the solid metal, thus in many cases simplifying the assembly of units. These materials have high mechanical strength, ductility and shock-resistance and offer good heat and corrosion resistance and permit fine filtration with low resistance to flow.

The porous metal finds application in the mixing of gases and liquids. Among the many varied uses may be mentioned: distributors for fermentation in yeast manufacture; production of cresylic acid where copper-nickel-tin distributors are employed.

### Shark Oil & Shark Skins

THE PRODUCTION OF SHARK LIVER oil, the skinning of sharks and curing of the hides, and the preparation of the fins for the market are described (*Fisheries Newsletter*, 1948, 7, 8).

For satisfactory oil extraction the fresh livers are minced and boiled with one-half their volume of water in a copper container placed in a water bath. The heat obtained by boiling the water in the outer vessel is enough to render the oil from the livers. The heating is continued for an hour at boil. On standing the oil floats on the surface of the mixture which is laddled off and strained through sacking to remove all solid matter. The clean oil should be stored in tightly sealed cans or drums.

The following method is recommended for skinning the

sharks: The opening is made along the back and not the belly. A very sharp knife is essential. Attempt should not be made to skin the hide off cleanly, but a good layer of flesh should be left on the hide. This flesh can be safely and easily removed in a subsequent process described as "fleshing".

The tail is cut off at the knob just before the tail fin starts; the other fins are then cut off. The knife is inserted in the holes made by removing the midback (dorsal) fins, and the hide is split along the middle of the back. Next, cuts are made around the gills and lower jaw.

The carcass is turned on its belly and the hide is peeled off starting from the left side—split off the hide near the head. The hide should not be pulled hard during peeling as tearing off the hide will produce "thin bellies". The skin should be held taut while peeling. The knife should always be employed to free the hide and tearing off should not be resorted to when the left side is skinned off; the right side is peeled off similarly.

After the hide is removed, it is washed thoroughly in sea water, removing all blood and slime and put in a barrel containing brine (7.5 lb. salt to 25 gal. of sea water). This soaking facilitates fleshing.

Fleshing can be done directly on the freshly-skinned hide, but it is more easily carried out after the hide has stood for 3 or 4 hr. in the brine solution. On no account should the hide be allowed to remain in this solution longer than overnight. Fleshing is done with a "beaming knife", a curved 16" blade with handles at each end. The hide is stretched on a beaming board, with a curvature matching that of the knife. The hide edges must be thoroughly flesh-free as this will assist in preserving the hide. Next, the tail end is split by cutting from the hole around the ventral fins through the hole left by the anal fins and then in a straight line to the end of the hide.

Curing is done on a platform with a slight slope so that the water and brine can run off. Salt is sprinkled on the inclined platform and the first hide is placed, flesh side up, flat on the top of it. There should be no creases or large folds in it. A generous amount of salt is spread over all portions and in particular salt should be rubbed along the cut edges. The

other hides are heaped up and treated in the same manner until a pile of 3' to 4' is obtained.

Hides take 3 to 5 days to cure and should not be allowed to stand for more than 6 days. The hides are cured in yards sheltered from sun, rain and heavy dew. If during or after curing the hides show a tendency to become pink, or if after curing they develop damp "weeping" spots, the trouble may be due to the salt employed and should be replaced. Once the pink colour makes its appearance a thorough disinfection of the curing platform is recommended.

The hides are packed for shipping after a fresh supply of salt is sprinkled on the hides.

Shark fins are commercially valuable and are in demand for making certain food specialties, such as Chinese soups. There is ready market for them in Singapore and China. The upper lobe of the tail is discarded and fins of sharks of less than 5' in length are of little value.

The fins should be cut off before skinning, cut correctly on a curve into fins and cleaned free from meat and skin. They are then soaked for a few hours in brine and final cleaning is finished. Care is taken not to leave large pieces of bone on the fins. The fins are dried in the sun spread over chicken wire and take about 14 days of good weather for drying. When properly dried they are stiff and hard.

#### New Uses for Herring Oil

NEW USES FOR SURPLUS HERRING oil are described (*Chem. Age*, 1948, 59, 790). The oil is employed for making margarine. The dissolved protein residue, left over after oil extraction, is dried in an equipment normally used for drying milk, whereby a protein powder is obtained. This can be employed as a valuable compressed food extract and animal feed.

#### A New Refining Process for Soyabean Oil

A NEW REFINING METHOD WHICH improves the flavour and quality of soyabean-salad oil has been developed in the laboratories of the *United States Department of Agriculture*. The process materially reduces the "painty", "fishy" or "grassy" flavour associated with the oil, and consists in the addition of citric acid—3 to 4 oz. per ton of oil. The additive is

said to function as a "metal scavenger" neutralizing the effect of minute quantities of metal that gets into the oil, mainly from the refining equipment; it helps to decrease the rate of oxidation of the oil, thus retarding the development of off-flavours.

The processed oil is said to taste better initially and keeps 3 to 5 times longer than soyabean oil refined by previous methods. The process is already in commercial use (*USIS*).

#### New Flotation Media for Mineral Recovery

HEAVY DENSITY LIQUIDS AND mixtures are normally employed in "sink-float" method of mineral separation and beneficiation. The main obstacle in employing heavy density liquids is their high price. These liquids can be substituted by suspensions of finely powdered heavy solids with water. Three ideal medium solids that fulfil the requirements, viz. ferrosilicon, galena and magnetite, are being used increasingly in sink-float plants in America. Some of the important characteristics and applications of these medium solids are described (*Chem. Age*, 1948, 59, 795).

Ferrosilicon is cheaper and harder than galena, easily recoverable by magnetic separators. The successful use of ferrosilicon-iron ores led to its use in the concentration of a variety of metallic and non-metallic ores—lead, zinc, tin, garnet, fluorspar, magnesite, baryte, spodumene and diamond ground.

Galena is heavy and can be recovered by froth floatation. It has the disadvantage of being so soft that it is abraded rapidly and lost as slime.

Magnetite being highly magnetic is easily recovered and is heavy enough to yield any gravity required in coal-cleaning. It costs only a fraction of the cost of ferrosilicon. It is somewhat easier to handle because of its lower specific gravity. It can be more easily ground than the other medium solids and for this reason forms a more stable medium. The fine size to which it can be ground reduces the wear on the equipment.

In addition to coal, magnetite can be employed as a medium solid for the separation of other low-gravity minerals such as gypsum, potash ores and concrete aggregate. Labour requirements for flotation plants are low and the separation capacities high. A unit



equipped with a 7' dia. cone separatory vessel can treat more than 600 tons of feed in 24 hr.

Although the use of commercial sink-float installations is restricted to the treatment of materials coarser than 10 to 14 mesh, pilot plant studies at the mineral dressing laboratory of *American Cyanamid Co.*, indicate the possibility of making good-gravity separations of particles as fine as 48 mesh in the case of ores and 100 mesh in the case of coal.

### Wood — Perennial Source of Organic Chemicals

VARIOUS WOOD-USING INDUSTRIES have in their hands the raw materials for the production and synthesis of a great number of organic chemicals useful in all walks of life. Many of these products are wasted at the present time, but fundamental research in these fields is uncovering new products and novel uses for the by-products of the wood-using industries.

Wood has one great advantage over coal and petroleum as a source of organic chemicals because, of the three, it is the only one that can be replenished in a relatively short time. Therefore, the day is not far off when wood will be one of the chief sources of organic chemicals.

In addition to the well-known chemical products of the wood-distillation industry (methanol, acetic acid and acetone) many new fine chemicals isolated directly from the wood distillate (such as  $\gamma$ -butyrolactone, *o*-cresol, guaiacol, 4-ethylguaiacol, pyrogallol, 1, 3-dimethyl ether, maltol, etc.) have appeared recently on the market in commercial quantities. These primary products open up possibilities of varied synthetic approaches.

The naval stores' industry based upon the extraction of pinewood stumps has evolved into a complex terpene chemical industry, with many new terpene and terpene derivatives on the market. The raw materials for many of these terpene processes are available also in the kraft-pulping industry's by-product turpentine and tall oil.

The sulphite pulp industry possesses in its sulphite waste liquor an enormous potential source of chemicals. The sugar content can be fermented to ethanol, butanol, acetone, lactic acid, or several other fermentation products, each of which may be employed in the synthesis of many other compounds. In fact, in

Sweden, sulphite liquor ethanol is the raw material for the country's organic chemical industry. Western hemlock sulphite waste liquor may be extracted directly to obtain conidendrin which is valuable for the synthesis of anti-oxidants.

The lignin in sulphite waste liquor can be oxidized to give high yields of either vanillin or vanillic acid. These interesting compounds are valuable for the synthesis of numerous derivatives which have found utility as non-toxic food preservatives, efficient sunscreens and sunburn preventatives, plasticizers for transparent film and cellulosic materials and therapeutic agents.

The hydrolysis of wood offers another source of sugars and fermentation products similar to those obtainable from the sugars of sulphite waste liquor (*Chem. Eng.*, 1948, 56, 299).

### Mobile Fuel Research Unit

TO PROVIDE FUEL CONSUMERS in industry with instruments and modern testing methods to improve fuel efficiency the British Ministry of Fuel has inaugurated a mobile testing unit service equipped with an extensive range of indicating and recording instruments. The unit visits works in any part of the country, to assist works' managements in obtaining, free of charge, full information regarding process heat requirements and maximum fuel conservation (*Chem. Age*, 1948, 59, 781).

The equipment is housed in an R.A.F. radar van specially built for transport of delicate instruments over rough ground. The van is fitted with detachable aluminium instrument panels for use either in the van or outside.

Instruments included are: a 6-point temperature recorder which can be used on three ranges: 0°-500°C, 0°-1,000°C. and 0°-1,600°C., two 2-point temperature recorders, a number of temperature indicators of various ranges with multi-point selector switches, a steam-flow indicator, a carbon-dioxide recorder and indicator, 2 steam-flow recorders—one using the normal mercury U-tube differential and the other an aneroid bellows type-differential, both provided with 3 range tubes to give full-scale deflection with 3 different flows.

A comprehensive range of orifice plates and carrier rings is carried covering pipe sizes up to 6" bore. A differential-pressure recorder

with a differential of 3" W.G. is provided for recording gas flow. The equipment also includes pressure recorders with a variety of ranges from a few inches W.G. to 500 lb./sq. in., recording and indicating draught gauges, a water-testing outfit, a workshop potentiometer, a millivolt meter, a multi-range ammeter, a kW. meter, a tachometer, a velometer, a surface pyrometer, a steam-engine indicator and chemicals and reagents, etc. The van is fitted with a fully equipped engineers' workshop and carries its own cable drums so that its switchboard may be coupled to a work's supply for power purposes.

### Chemical Industry in Ceylon

THE WIDE SCOPE FOR CHEMICAL industries, organic and inorganic, existing in Ceylon is reviewed (*Chem. Age*, 1948, 59, 793).

The possibilities of utilizing waste tar are being investigated in a plant built for the purpose in the laboratories of the Department of Industries, Colombo. The obvious by-products include disinfectants, wood preservatives, aspirin, alcohols, ammonium sulphate and naphthalene. The residual tar can be used for road-surfacing.

The Department of Industries has worked out a process for recovering by-products from coconut shell-distillation industry. Wood naphtha and two brands of wood preservatives have been produced.

Ceylon has extensive natural resources for the manufacture of soap and perfumes on a large scale. Coconut oil and citronella and cinnamon leaf oils are available in abundance.

The question of utilizing the iron ore deposits, found in the Ratnapura district and in parts of the Uva Province, is being investigated.

### "Safety First" in Laboratory Work

SOME PRACTICAL CONTRIBUTIONS to the information on safety measures in chemical work were made at a recent conference held in London by the *Institute of Physics* in collaboration with the *Institution of Metallurgists* (*Chem. Age*, 1948, 59, 688).

Mr. Idris Jones, of the *Chemical Research Laboratory (DSIR)*, pointed out that 16 per cent of the accidents were attributable to apparatus as distinct from human failings. The fundamental factor, he stressed, was the human one.



Instruction in safety methods should, he considered, be as part of chemical education. At the *Chemical Research Laboratory* a safety organization has been set up and a senior member of the staff is entrusted with the task of inspection and recommendation of safety measures. The Chemical Research Laboratory Safety Officer has drawn up a pamphlet on laboratory safety measures which has been published for public use.

Many of the speakers stressed on the cleanliness and orderliness to be maintained in the laboratories. Some of the measures under this head are: floor obstructions to be removed, rubber gloves to be kept on racks, metal parts aluminium-painted, transparent cases shielding balances, testing equipment and vacuum pumps, Buchner flasks and vacuum desiccators to be provided with metal guards and distillation units with expanded metal guards; for cold traps, the use of liquid nitrogen is recommended in place of air; cylinders to be used in a vertical position, in stands and chained securely; electrical connections should be kept short; pipettes used in measuring to be always used with an interposed bulb.

Certain guiding principles in organizing work in laboratories handling explosives were suggested by Mr. G. C. Ellington. The basic precaution to be taken in testing materials are: use of minimum amounts, transportation and handling of samples to be reduced to a minimum and special care to be taken in selecting the nature of the test (such as in determining moisture content by heating).

The safety measures to be taken in laboratories handling radioactive material are: the laboratory itself should be in a one-storey building, spaciouly laid out, allowing 8'-10' per operator; electrical fittings should be flame-proofed and gas supplied in iron pipes or armoured pipe; sinks should be numerous and floors unpolished; steam-heating is preferable to electrical and, where a continuous water flow was needed, its out-flow should be visible.

Precautions to be taken in atomic energy establishments were given by W. G. Marley. To avert the risk of dangerous exposure of the body to external radiation the intensity of radiation should be measured. The *National Physical Laboratory* runs a "film badge service", a badge of X-ray film worn on the exterior of clothing to indicate the maximum dose of radiation. Periodical surveys of the laboratory for local contamination should give geiger counter records of less than 500 per min. Removable bench tops, stainless steel-working trays, protective clothing, rubber gloves and over-shoes have been recommended.

### Announcements

*The Elliott Prize for Scientific Research* in Chemistry for the year 1949 will be awarded to the author of the best paper giving the results of original research carried out in Chemistry and published during the years 1945-48.

The prizes for the next four years will be offered as follows: 1950, Physics; 1951, Geology and Biology (including Pathology and Physiology); 1952, Mathematics; 1953, Chemistry. The papers should be submitted to reach the President, *Royal Asiatic Society of Bengal*, 1 Park Street, Calcutta, by the end of June each year.

*National Institute of Sciences of India* — At the annual general meeting of the *Institute* held at Allahabad in January 1949, the following office-bearers were elected: Prof. S. N. Bose (*President*), Prof. A. C. Banerjee and Maj.-Gen. S. S. Sokhey (*Vice-Presidents*), Dr. J. N. Mukherjee (*Foreign Secretary*), Prof. D. S. Kothari and Dr. H. S. Pruthi (*Secretaries*).

Dr. D. D. Kosambi of the *Tata Institute of Fundamental Research*, Bombay, has been awarded a *UNESCO Fellowship* tenable in U.S.A. to work on new types of electronic calculating machines for setting up a modern calculating laboratory in Asia. Dr. Kosambi has been invited as a visiting professor by the Chicago University. He is also to serve on

a Committee of mathematicians to select the recipients of Field Medals to be awarded at the forthcoming *International Congress of Mathematics* to be held at Massachusetts.

### Erratum

*Indian Institute of Chemical Engineers* — The correct address of the *Institute* is 5/2, Garstin Place, Calcutta 1, and not 5/2, Garlic Place, Calcutta 1, as reported previously.

## REPORTS FROM STATES & PROVINCES

(continued)

last 8 years. The use of steam and modern machinery for the manufacture of sugar candy, and the utilization of the mother liquor for the manufacture of high grade confectionery, are the important aspects of the processes developed. The cost of production has been appreciably reduced and the output of sugar candy enhanced. It has been possible for the first time to produce bright, lustrous, large and well-formed candy crystals in a specially designed rotary candy crystallizer.

Advice will be given, on application, to interested persons in matters relating to processing, equipment and quality control in the manufacture of candy and confectionery. Training is also imparted to interested chemists. The training period is of one month duration, and usually starts from January each year.

### J. K. Institute of Applied Physics

THE FOUNDATION-STONE OF *Institute* was laid by Pt. Jawaharlal Nehru in the Muir College grounds, Allahabad, during the last Indian Science Congress Session. Sir Padampat Singhania has donated Rs. 5 lakhs towards the establishment of the *Institute* for conducting research work in Geophysics and Radiology.

# Reports from States & Provinces

## BOMBAY

### Industrial Finance

#### Corporation

THE GOVERNMENT HAVE DECIDED to set up a finance corporation to assist medium, small-scale and cottage industries in the Province. The corporation, which would be a statutory body, will grant loans to medium and small-scale industries and will have a special department for handicrafts and cottage industries. 51 per cent of the total capital will be subscribed by the Government.

## CENTRAL PROVINCES

### Newsprint Factory

THE PREMIER OF C.P. AND Berar laid the foundation-stone of the *National Newsprint & Paper Mills* at Chandni in Khandwa district on December 26. The mill is the first newsprint factory in the country, and is expected to start work by the end of 1949. It will manufacture 100 tons of newsprint per day.

### Coal-mining

PRELIMINARY INVESTIGATIONS have been completed by the Government on the working of coal mines at Khorba Drug in district, 23 miles from Champa station, on the Bilaspur-Calcutta railway line as a State enterprise. The investigations were initiated with a view to determine the extent of metallurgical coal deposits that could be profitably utilized in setting up of an iron and steel factory in the vicinity of the coal-mining area.

Coal has also been located close to the Khaparkheda Thermal Station, Chhattisgarh district, which is calculated to produce 30,000 kW. This field will also be run by the Government.

## MADRAS

### Conference of Steam Users & Boiler Inspectors

A CONFERENCE OF STEAM USERS and Boiler Inspectors of the

Province was held recently at Madras to consider the difficulties in the working of the Indian Boilers Act and to discuss problems of availability and economic utilization of fuels.

Mr. Campell, the Chief Engineer of B. & C. Mills, Madras, pointed out that some of the existing steam plants in the Province were able to utilize only 20 per cent or even less of the heat available and the rest was wasted through inefficient methods of utilization. He endorsed the view of the Chief Inspector of Boilers that advantage should be taken of the economic use of back pressure machines to obtain larger blocks of power as a by-product of the process steam. He also pointed out that some steam users in the Province had installed such steam plants but factory owners' efforts to increase the efficiency of their plants were thwarted, to some extent, by the Government Electricity Department in so far as the present needs of the Department did not permit industrial generating plants to run in continuous parallel operation with Government electric system. This meant that thousands of tons of fuel which could be used for many essential purposes now requiring it were wasted. This matter should receive the earnest consideration of Government.

### Fruit Farms

THE OPENING OF FRUIT FARMS AS adjuncts to the 6 Agricultural Research Stations in the Province has been sanctioned by Government. About 10 acres of land will be utilized for the purpose at each station, except at Pattambi where only 5 acres would be allotted for fruit cultivation. The Government have sanctioned Rs. 5,000 for improving irrigation facilities at Gutur Station for work on orchard methods.

### Fishing Industry

A REVENUE OF RS. 14.73 LAKHS was realized by the Government during 1946-47 from the Fishery Department. As a result of

deep sea-fishing operations initiated by the Government in 35 centres in the Province, a total of 745,960 lb of fish was caught during the year.

The total quantity of crude shark liver oil received at the *Government Oil Factory*, Calicut, from the curing yards and oil extraction centres was 75,591 lb. The quantity of standard oil produced was 93,600 lb. and 102,087 lb. of oil was sold during the year.

## Pennar-Kumudhvatthi

### Project

THE GOVERNMENT WILL TAKE UP the execution of the project this year. A sum of Rs. 19 lakhs has been sanctioned for the work which envisages the harnessing of the Pennar river in the Kurnool district in Madras and the Kumudhvatthi river in Mysore by constructing a dam at the confluence of the two rivers. The project will ensure a perennial supply of water for irrigation purposes and will bring under cultivation about 3,200 acres of virgin land.

## NEW DELHI

### Land Reclamation

A FIVE-YEAR PLAN TO RECLAIM 6 million acres of uncultivated land by heavy tractors is under the consideration of the Central Ministry of Agriculture. The results of reclaiming 36,000 acres of land in the United and Central Provinces have been encouraging; the costs incurred in their cultivation are reported to have been covered by the bumper crops raised. This plan is expected to increase the country's annual foodgrain production by 2 million tons.

## UNITED PROVINCES

UNDER THE AUSPICES OF THE *Indian Institute of Sugar Technology*, Kanpur, arrangements have been made for training technicians in the manufacture of sugar candy and confectionery by improved scientific methods. Details of the course and the prospectus can be had, free of charge, from the Chemist-in-Charge, Sugar Candy Research Scheme, Ravalgaon, Dt. Nasik, C.P. & Berar.

The Sugar Candy Research Scheme has been working under the guidance of the *Indian Institute of Sugar Technology* for the

(continued on page 110)

# INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the *Gazette of India*, Part II, Section I, for January 1949.

## Inorganic Chemicals

39754. NORANDA MINES LTD. : Process of recovering sulphur from pyrite : *Passing a gaseous mixture containing oxygen in an ignited mass of pyrite.*
38164. I.C.I. LTD. : Production of sulphuric acid or oleum : *Converting molten sulphur into sulphur dioxide up to 14 per cent and converting sulphur dioxide into trioxide.*
39992. I.C.I. LTD. : Indication of ammonium nitrate (Addition to No. 38787) : *Mixture of 100 parts of ammonium nitrate and 0.01 to 1.0 parts of sulphonated aromatic dyestuff other than acid Magenta.*
36767. Moos : Utilization of the waste product of calcium chloride industry, viz. hydroxide pastes : *Treating the waste product of magnesium hydroxide paste with sulphuric acid.*
39686. UNITED STATES RUBBER CO. : Recovery of fluosulphonic acid : *Admixing sulphur trioxide with spent acid and distilling the mixture.*
39957. ZINC PRODUCTS LTD. : Improved process and apparatus for the manufacture of zinc oxide : *Molten zinc maintained below boiling temperature and oxidation of zinc promoted by increasing the surface of molten metal.*
37972. BRITISH PERCLASE CO. LTD. : Improvements in the manufacture of magnesium hydroxide : *Slaking lime from dolomite and adding it rapidly to magnesium compounds.*

## Organic Chemicals

40610. CIBA LTD. : Manufacture of a new condensation product of 6-sulphanilamido-3-methylpyridazine : *Comprises reacting 6-sulphanilamido-3-methylpyridazine with formaldehyde.*
37706. I.C.I. LTD. : Manufacture of new dyestuffs of the anthraquinone series : *Halogen atoms of compounds of anthraquinone series and related types carrying halogeno methyl groups are replaced by quaternary or ternary salt groups.*

## Miscellaneous Chemicals

39694. STANDAERT & BROWN : Production of double and triple superphosphates and of compound fertilizers containing the same : *Double or triple superphosphate is blended with nitrogenous and/or potash fertilizers.*
38014. I.C.I. LTD. : Manufacture of sulphuric esters of leuco derivatives of vat dyestuffs and anthraquinone intermediates : *Reacting corresponding leuco compound or its metal salt with sulphur trioxide in presence of an organic amide.*

## Food & Kindred Products

40018. EAST ANGLIA CHEMICAL CO. LTD. : Production of dextran : *An aqueous extract of fresh*

*yeast of low solids content is used for the production of dextran from sucrose.*

40392. LEVER BROTHERS & UNILEVER LTD. : Improvements in the catalytic treatment with hydrogen of glyceride oils : *Hydrogenating the oil above 150°C. in the presence of non-colloidal particulate nickel-alumina-silica catalyst having between 15 and 50 per cent by weight of nickel.*

## Instruments

37235. KHORANA & BHUNVARA : Vacuum operated automatic burette : *Providing a two-way stop-cock between one arm of a T-tube connected to reservoir and the side tube from the top of burette.*
37622. TECALEMIT LTD. : Improvements in air separators : *The air separator has diaphragms to divide the liquid into a number of shallow superimposed streams and is fitted with an air vent valve at the top.*

## Metals & Metal Products

38621. JOHN MILES & PARTNERS (LONDON) LTD. : Improved process of reducing metals from their ores : *Reducing ore by hydrogen and smelting and refining the reduced ore by oxygen : hydrogen and oxygen being obtained by electrolysis of water.*
37824. H. J. ENTHOVEN & SONS LTD. : Soldering : *Soldering in presence of a flux consisting of a hydrazine-hydrohalide.*
40209. SINGH : Manufacture of cast irons : *Treating molten cast iron with magnesium before casting.*
38346. PARANJPE : Production of sound castings : *Mould cavity exhausted by creating vacuum, maintaining the vacuum until metal solidifies.*

## Physics (General)

34695. CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE : Production of nuclear energy : *Comprising uraniumous mass inside which are placed damping masses formed of at least one neutron slowing down substance.*
34477. CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE : Production of nuclear energy : *Production of energy through nuclear fission of uranium element in a form in which isotope 235 is in a higher proportion by means of chain reactions.*

## Miscellaneous

38990. BURNDIPT LTD. : Improvements in primary electric cells : *Plastic sheathing wrapping container sealed at edges and a perforation in container clear of electrolyte.*

## Eclecticism in Medicine

THE report of the Committee on Indigenous Systems of Medicine, recently published by the Ministry of Health, Government of India, is a stimulating document based on the detailed inquiries into the potentialities and achievements of the systems of medicine serving the people of this country. In an article entitled *Rationalization of Medicine in India*\* appearing in these columns, we have referred to the practicability of bringing together the indigenous and modern systems of medicine, and the desirability -- nay, imperative necessity -- of utilizing the services of large numbers of practitioners of systems indigenous to this country for building up an adequate national health service. This was the thesis of the address of Col. Sir Ram Nath Chopra to the 1948 Session of the Indian Science Congress (Patna). The views expressed by him have been further emphasized and amplified by the Committee on Indigenous Systems of Medicine, over which Col. Sir Ram Nath Chopra presided.

The Committee was appointed in December 1946 to meet the public criticism which the Bhoré Committee's failure to assess the value of indigenous systems of medicine evoked, and the Committee was charged specifically "to consider and recommend the steps to be taken to improve facilities for research in indigenous systems, and generally to increase their usefulness to the public". The Bhoré Committee had reported: "We are unfortunately not in a position to assess the real value of these (indigenous) systems of medical treatment as practised today, as we have been unable with the time and opportunities at our disposal to conduct such an investigation into the problem as would justify clear-cut recommendations", and this after recognizing that the Western medicine, as practised in India, provided at

best only a part of the country's requirements for medical relief, and that doctors trained in allopathy were concentrated in urban areas "so much so the proportion of doctors to the rural population was such as to result in a total denial of effective medical aid to the people". The aid available to the masses in India is that provided by the *Ayurvedic* and *Unani* practitioners, and the people have shown a preference for these systems which have their roots in the soil. The indigenous systems are "time-tried and are based on sound observations, and many of their theories are capable of withstanding scientific investigation". Despite long and liberal State patronage, the Western system has failed to penetrate into the rural areas and minister to the needs of the vast majority of the people. This is not to minimize the value of the Western system, or to state that all is well with the practice of indigenous systems. The neglect from which the latter have suffered for centuries has resulted in the accumulation of a mass of superstition and dogma, and the incursion into them of a large number of ill-qualified and ill-equipped practitioners. In this respect their position is not different to what was obtaining in the Middle Ages in Europe when the classical system of medicine as taught by Hippocrates and Galen had degenerated into superstitious beliefs and quackery. In its development, allopathy has drawn freely from the knowledge available in the East, and thanks to the labours of a large body of savants working in well-equipped research laboratories all over the world, it has attained a position of pre-eminence. The indigenous systems, on the other hand, have stagnated and have fallen from their pristine glory, and their practice has suffered from the "human errors" which, according to Roger Bacon, arise from undue respect for authority, habit, prejudice and false conceit of knowledge. The first thing, then, is to rid

\* March 1948, p. 101.

the systems of these "errors" and to vitalize them by the methods of experiment and observation. Research should be directed "(i) to clear these systems of accretions of centuries of doubtful value; and (ii) to give scientific meaning and significance to the fundamentals of these systems so that they may be accepted by science".

"Truth and science are one", said H.E. Sri C. Rajagopalachari in his reply to the address presented to him by the *Bangiya Ayurveda Mahasabha* in Calcutta (June 17, 1948). "There can be no competition between truth and truth, but only between truth and error. I would, therefore, entreat you to remember 3 things: The first is to demand, and not oppose, a high standard of education and equipment and careful selection in admitting students to your colleges for courses in medicine. The second is to include modern scientific knowledge alongside the traditional *Ayurveda* in your institutions so that truth may run in a single course and prejudice and ignorance vanish to the minimum point. Thirdly, scientific research should be encouraged and there should be no opposition but full co-operation in this between Western doctors and the learned *kavirajas*." This sage advice has been accepted by the Committee which "does not believe that there can be separate systems of Western or Indian medicine. Science is universal and medical science is no exception to this rule. Such multiplicity of systems is only believed and encouraged by people who have not clearly grasped the significance of the noble ideals as preached by the great *Acharyas* of Indian medicine and the savants of Western medicine. The so-called systems merely represent different aspects and approaches to medical science as practised in different ages and in different parts of the world. The aim of all systems is the maintenance of health and prevention and cure of disease. Anything of value emerging from them should be integrated and utilized for the benefit of humanity as a whole without any reservations." Starting from this incontrovertible premise, the Committee proceeds to collect and appraise evidence—oral and documentary—from leaders of Western and indigenous systems, and to examine critically the reports of Committees appointed by Provincial and State governments to inquire into, and report upon, the facilities for education and research in indigenous systems as obtaining in their respective regions. The result is a unanimous report embodying

valuable observations and recommendations for orienting medical education in the country with a view to integrating the different systems, and for rationalizing medical practice in the country as a whole. The Report observes "Indian medicine is defective in the field of surgery, obstetrics, structural physiology and pathology, and must look for help to Western medicine in these branches. On the other hand, great emphasis is laid on the prevention of disease by improving the 'soil' factor by adopting suitable dietary which has been elaborately worked out according to seasons and climates and the temperaments and avocations of individuals. Western system has yet to formulate a coherent system of principles of medicine and is in marked contrast to Indian medicine in this respect. Western and Indian medicines have thus much to give to each other, and a proper synthesis of the two will benefit humanity as a whole." The Report points out that such a synthesis "though not easy" is not only possible but practicable and essential. "It must, however, be admitted that its successful working will neither be easy nor quick. It will require the continued and painstaking research of many first-class experts for many years to evolve a complete integration that could be of real value to India in particular, and the world in general." How this research is to be organized and carried out has been fully dealt with and the steps which have to be taken immediately to make "a beginning in the right direction" have been indicated.

The emphasis placed on the "integration" of apparently different systems of medicine has given rise to the fear, in certain quarters, that the acceptance of the Committee's recommendations would be tantamount to bringing pressure on Western medicine to compromise with principles. It is feared that a "hybrid" system would result which may prove disastrous. A dispassionate study of the Report, however, reveals that the Committee does not call for any compromise with principles. What the Committee seeks to achieve is "a wise eclecticism with the main emphasis on the Indian system". This is sought to be achieved not by compulsion, but by the pooling of knowledge wherever it is found, by objective study and research. It is realized that the desired result cannot be obtained quickly or easily. Years of research by large numbers of scholars versed

in the many schools of medicine would be necessary, and the outcome of their labours would benefit not any one particular system but medicine as a whole.

The Report envisages the establishment of a Central Research Institute of the uni-purpose type. On the need for research on the subject listed in the Report there will be general agreement, but it is desirable, even necessary, to consider whether the Central Drug Research Institute, established by the *Council of Scientific & Industrial Research* at Lucknow, would not serve the purpose which the Committee has in view. An expert committee of the *Council of Scientific & Industrial Research* has thoroughly examined the problems of medical research in India, and the departments to be established at the Lucknow Institute are mostly those proposed for the new Central Research Institute. It would be prudent to consolidate, strengthen and

develop established institutions rather than to add to their number, an addition being justified only if it is established, as a result of a thorough inquiry, that one or the other of the existing institutions cannot be made to serve the needs in view.

The Committee's approach to the problems of medical education and research is objective and scientific, and discloses a width of outlook which is at once refreshing and stimulating. Its recommendations on the organization of medical relief in the country are characterized by realism. The Committee may well be proud of having found "a reasonable, practical and economical approach to the problem of medical relief to the people of this country in general and rural areas in particular without a violent disturbance of the existing state of affairs". We feel sure that their plea that the scheme may be given a fair trial would find wide support in this country.

## World's First Dictionary of Colours

BRITAIN IS PRODUCING A UNIQUE AND UNUSUAL work of reference. It is a *Dictionary of Colours for Interior Decoration* and will be the first of its kind ever to be published. The dictionary will contain standard specimens of 378 different colours. These are illustrated on both plain and matt surfaces as well as on pile fabric. Each carries its own individual reference number.

This reference volume should prove invaluable not only to dyers and manufacturers of paints, fabrics and pottery, but also to the ordinary householder. It will eliminate all confusion and debate over the nomenclature of various shades of any one colour. Also, by the use of the standard reference numbers, it will make it possible to ensure perfect matching of shades either in interior decoration schemes or in dress fabric. (*B.I.S.*)

## Dr. James William McBain, Director, National Chemical Laboratories, Poona

**D**R. James W. McBain, Professor of Chemistry emeritus at Stanford University in California, recently accepted appointment as Director of the National Chemical Laboratories in Poona. The appointment is for three years, beginning in October 1949.

Born in Chatham, New Brunswick, Canada on March 22, 1882, Dr. McBain received his Bachelor of Arts degree from the University of Toronto, Canada, in 1903, and his Master of Arts degree a year later. He was a student at the University of Leipzig for two years, and received a Doctor of Philosophy degree from Heidelberg University in 1906.

Known as a world authority on soap, McBain's interest in this particular field began when he was a student at Heidelberg. McBain's researches on soap have contributed a vast amount of knowledge in the field of physical chemistry and at the same time has had wide practical application.

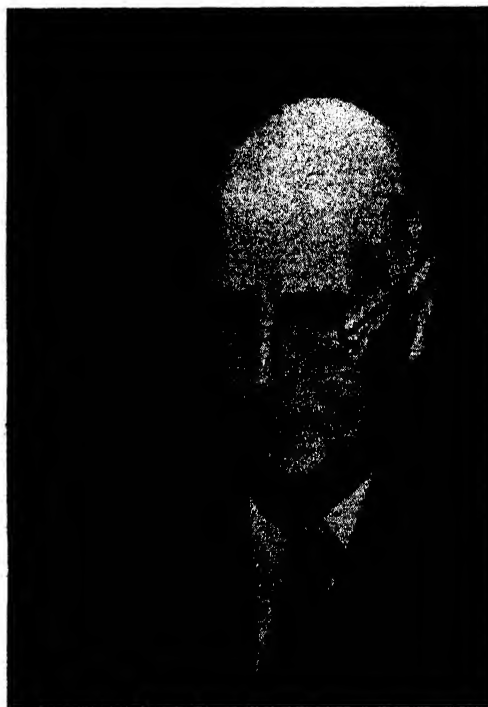
During World War II, Dr. McBain did much vital war work at Stanford and his specialization was particularly important in certain aspects of synthetic rubber manufacture, in the problem of foaming of aircraft oils, and in the thickening of fuel for incendiary bombs and flame-throwers. This was the second time Prof. McBain had been engaged in such war work; during World War I, when he was serving with the British Army in France, the British Munitions Ministry had called him back to England to do research in poison gases and explosives.

Since 1947, when Dr. McBain became Professor of Chemistry emeritus at Stanford, his "retirement" has consisted of continuing research and scientific writing. In addition, he has supervised two Office of Naval Research contracts at the Stanford Research Institute—one on the depth of a surface liquid which led to important new discoveries concerning the molecular make-

up of liquid surfaces, the other on the nature of bubbles in aqueous solutions.

Prof. McBain's publications in physical chemistry make a shelf of heavy volumes—he has published nearly 400 scientific papers on various topics in that field.

He has been a councillor and vice-president of the Faraday Society, is a Fellow of the Royal Society, and was awarded that organization's Davy Medal in 1939. He is a member of the National Institute of Social Sciences, the American Chemical Society, the Bunsen Gesellschaft, the British Chemical Society, the Society of Rheology, the American and British Associations for the Advancement of Science, and Colloid and Structure of Liquids Committees of the National Research Council (*USIS*).



DR. J. W. MCBAIN

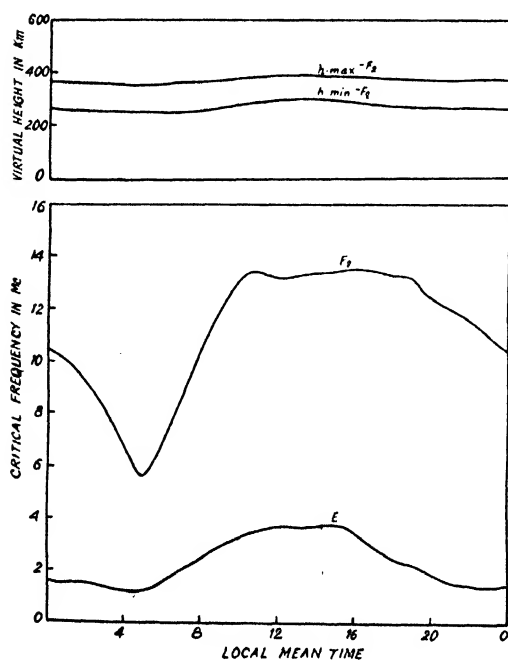
# Characteristics of the Ionosphere over Calcutta (January 1949)

S. S. BARAL, S. N. GHOSH, R. K. MITRA,  
D. C. CHOUDHURY & (Miss) T. K. PAL

*Wireless Laboratory, University College of Science, Calcutta*

THE following are the ionospheric data observed at Calcutta for the month of January 1949. The observations were made at each hour of the day for 5 days a week.

Fig. 1 presents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in the graphical form during the month of January 1949. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during April 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when E region ionization was found to be abnormal and the



(5 HOURS 54 MINUTES AHEAD OF G.M.T.)

FIG. 1 — JANUARY 1949.

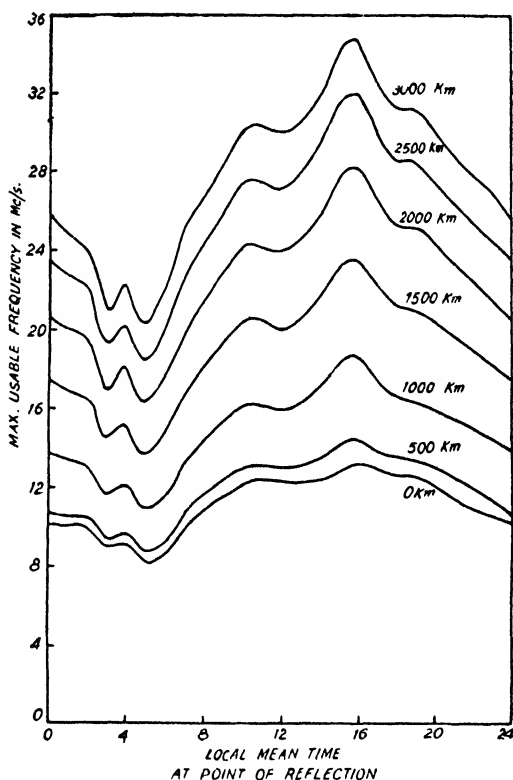


FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION VIA  $F_2$  LAYER, APRIL 1949.

corresponding penetration frequencies and heights. The frequency of occurrence of abnormal E ionization was high during some nights and also during the afternoon on some days.

During the latter part of this month there was high sun-spot activity and day-to-day observations show that on some days the region F attained great heights and thicknesses during daylight hours and high ionization on the corresponding nights.



TABLE I

MONTH & YEAR	DATE	HOUR	$f_{E_s}$	$h_{E_s}$
			Mc.	Km.
Jan. 1949	3	17.00	3.30	120
		18.00	3.35	120
		21.00	3.40	120
		22.00	3.40	120
		23.00	2.85	120
	4	03.00	2.40	120
		04.00	4.05	120
		05.00	4.45	120
		06.00	3.05	120
		07.00	3.20	120
	5	08.00	3.20	120
		17.00	3.50	135
		19.00	2.15	120
		20.00	2.80	120
		21.00	2.75	120
	6	22.00	3.00	120
		23.00	3.00	120
		00.00	2.00	120
		07.00	2.00	120
		08.00	3.25	120
	7	16.00	4.45	135
		17.00	4.25	135
		19.00	2.00	120
		20.00	2.35	120
		21.00	2.20	120
	8	22.00	2.20	120
		23.00	3.00	120
		00.00	2.15	105
		02.00	2.00	105
		13.00	5.15	135
	10	14.00	5.20	135
		15.00	5.20	135
		16.00	5.20	135
		17.00	5.00	135
		18.00	2.60	120
		19.00	2.10	120
		20.00	2.30	120
		22.00	2.10	120
		23.00	2.65	120
		00.00	2.25	120
		04.00	2.20	105
		17.00	3.10	120
		18.00	2.40	120
		19.00	3.00	120
		20.00	2.50	120
		21.00	2.10	120

TABLE I—contd.

MONTH & YEAR	DATE	HOURS	$f_{E_s}$	$h_{E_s}$
			Mc.	Km.
Jan. 1949	11	01.00	2.20	105
		02.00	2.20	105
		08.00	3.45	120
		09.00	3.35	120
	17	15.00	5.20	135
		16.00	4.20	135
		17.00	4.70	135
		18.00	2.30	120
		21.00	3.25	120
	18	23.00	6.00	190
		01.00	5.20	120
		02.00	3.50	120
		03.00	2.60	120
		04.00	2.35	120
	20	06.00	2.30	120
		10.00	3.95	120
		17.00	3.20	120
		18.00	2.50	120
		20.00	2.70	120
	21	21.00	3.30	120
		22.00	2.70	105
		23.00	2.70	105
		00.00	2.30	105
		03.00	2.60	105
	27	04.00	2.50	105
		05.00	2.50	105
		08.00	2.60	120
		18.00	3.55	120
		19.00	3.00	120
	28	21.00	2.45	120
		22.00	2.25	120
		23.00	2.20	120
		00.00	2.05	105
		07.00	2.20	120
	31	08.00	3.05	120
		18.00	3.20	120
		21.00	3.30	120
		22.00	3.35	120
		23.00	2.40	120

## Opium Alkaloids—Recent Development in India

B. K. MUKHOPADHYAY

*Opium Agent, Ghazipur*

OPIMUM provides India with a raw material for the manufacture of some of the most important alkaloids used in medicine. The drug has been produced in our country from time immemorial and has been used as a medicine and also to a considerable extent as an intoxicant.

The invaluable medicinal properties of opium were recognized early by modern medical science. It was found out later that the essential properties of the drug are attributable to various basic substances contained in it, the most important of which are morphine and codeine, the latter being

constitutionally the methyl ether of the former. The use of the opium alkaloids, in the form of their soluble salts, in place of the crude drug itself has been adopted largely in modern medical practice because of the accuracy attainable in adjusting the dosage, adaptability of administration through injections, ease of handling and storage and other advantages.

Morphine is the most valuable remedy for pain and consequent sleeplessness. Its use (in the form of injections of its salts in solution) is now firmly established for the treatment of internal haemorrhage. Morphine has a specific action on the alimentary canal, diminishing peristalsis, and is employed in the treatment of diarrhoea and colic. In the treatment of insomnia of cardiac disease, morphine is extremely valuable because it does not affect the heart and peripheral circulation.

Codeine is valuable in allaying cough, specially the cough of phthisis, and is useful also in insomnia, where the sleeplessness is due to incessant coughing. Equally with morphine, codeine decreases sugar in the urine of diabetic patients owing to its effect on sugar metabolism, but is not used in the treatment of diabetes. Habit formation from its continued use is almost unknown and it is used in the withdrawal treatment of morphine addiction.

The quantity of morphine and codeine used for medicinal purposes annually throughout the world has been estimated at about 1,48,000 lb<sup>1</sup>. Medical treatment on modern lines, however, is as yet available to only a fraction of the world's population. The demand for the drugs, therefore, is sure to increase, as medical facilities are extended to hitherto undeveloped countries.

In India opium is produced solely under Government control (annual production C. 7,000 maunds). The major portion is used to prepare what is known as "Abkari Opium" or "Excise Opium", i.e. opium for sale to the public for use as a household remedy and also for consumption by habitual addicts. Opium smoking is now illegal in India. The drug is either made into pills for swallowing or drunk after being mixed with water. A portion of the raw opium is specially made into "medical" opium cake and powder corresponding to E<sup>2</sup>, specification for opium and *opium pulvaratum* respectively for sale to licenced druggists and pharmacists. Opium unfit for use for these purposes and waste opium is used for recovery of alkaloids in the *Alkaloid Works* attached to the *Government Opium Factory* at Ghazipur. The quantity produced is restricted at present to what is necessary to meet the demand in India, viz. some 1,500 lb. of the principal alkaloids.

The principal alkaloids present in opium are morphine and codeine. In Indian opium the amounts are roughly 10 per cent of morphine and 2 per cent of codeine. Of the minor alkaloids, thebaine may be mentioned. This is present to the extent of some 0.5 per cent in Indian opium. Authoritative literature<sup>2</sup> mentions that Indian opium does not contain papaverine, another opium alkaloid found to the extent of up to 1 per cent in Turkish and Persian opiums. Recent work, however, carried out under the writer at the *Alkaloid Works* at Ghazipur has definitely established the presence of the alkaloid in Indian opium, though the quantity is only about 0.1 per cent. The details of the work are being prepared for publication.

AGENCY	SUB-DIVISION	M.S. max.	M.S. min.	M.S.	M.S.	M.S.	M.S.
				10% & above	9-10%	8-9%	below 8%
PERCENTAGE of challan							
Banaras	Fyzabad	12.15	8.33	52.4	33.3	14.0	nil
	Ghazipur	11.79	8.39	46.7	26.7	26.7	nil
	Budaun	12.09	8.45	75.2	17.1	7.6	nil
	Bareilly	12.09	7.88	71.7	19.6	7.6	1.1
	Gonda	11.79	8.28	68.6	15.2	24.2	nil
	Bara Banki	11.85	7.70	49.3	26.1	23.2	1.3
	Azamgarh	12.09	8.39	80.6	11.1	8.3	nil
Malwa	Mandsaur	12.15	8.22	66.2	23.1	10.8	nil
	Indore	11.85	8.17	61.3	23.8	15.0	nil
	Partabgarh	12.09	8.33	74.4	17.9	7.7	nil
	Jasor	11.74	8.56	63.3	30.6	6.1	nil
	Sitaman	11.85	8.75	75.8	18.2	6.1	nil
	Neemuch	11.74	8.39	58.8	27.5	13.8	nil
	Zhalawar	11.53	8.81	56.5	34.8	8.7	nil
	Ratlam	11.74	8.92	50.0	37.5	12.5	nil
	Sailana	10.11	8.56	25.0	50.0	25.0	nil
	Piplaudha	10.45	8.75	20.0	60.0	20.0	nil
	Dewas	11.91	8.75	50.0	nil	50.0	nil
	Nimbahira	11.60	8.28	27.3	36.4	36.4	nil

Another alkaloid, narcotine, occurs in Indian opium to the extent of some 5 per cent. No use has yet been found for this product in quantities sufficient to justify regular production. The table above would give an idea of the amount of morphine present in Indian opiums.

For the recovery of morphine and codeine from opium the "Gregory" process<sup>3</sup> is adopted in India. The opium is exhaustively extracted with cold water, the extracts combined and concentrated and then treated with calcium chloride which converts the alkaloids into their soluble hydrochlorides. The solution is filtered and then evaporated to crystallization. The crystals are centrifuged off the mother liquor and then treated with excess of caustic soda solution, which converts the morphine into the soluble sodium salt, and separates the codeine as the base. The latter is then separated by repeated extraction with benzene and finally recovered after distilling off the solvent and purified. It is also converted into codeine phosphate or any other salt required. From the alkaline solution, morphine is precipitated by addition of ammonium chloride. The crude morphine is filtered off, redissolved in hydrochloric acid, treated with activated charcoal and filtered, the process being repeated until the solution is sufficiently colourless. Afterwards, it is either evaporated to crystallization to produce morphine hydrochloride, or the pure base is reprecipitated with ammonia, filtered off and converted into the sulphate or any other salt. The method followed in Germany is to extract the raw opium with methylene chloride which removes the narcotine and papaverine. The residue is treated with lime water and filtered, whereupon the morphine and codeine pass on to the filtrate as the calcium compounds. They are separated as usual and made into the various salts and derivatives. The procedure is not suitable for adoption in India, because the solvent methylene chloride is not manufactured locally and is difficult to obtain in sustained and regular supply from overseas.

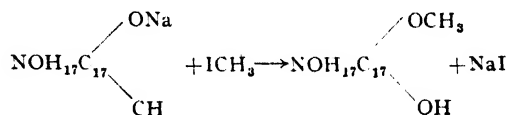
Owing to the greatly increased demands for morphine salts and codeine during the last war, the *Government Alkaloid Works* was equipped with modern machinery and fittings for recovery of the alkaloids from opium on a large scale. The plant, however, could be brought into use only after the cessation of hostilities. The capacity of the *Works* is now such that the present annual demand

of the Indian market, viz. about 1,500 lb., could be produced in less than two months.

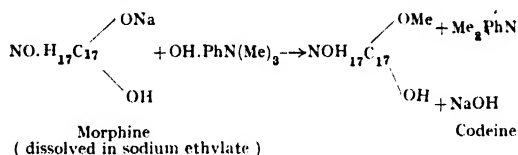
The world demand for codeine is considerable. Indian opium contains on the average about  $\frac{1}{3}$ th as much codeine as morphine. The proportion is much lower in Turkish and Persian opiums. The consumption of codeine, however, is considerably more than that of morphine. Therefore, large quantities of codeine are also produced synthetically.

The synthetic production of codeine consists essentially in methylation of morphine. The latter contains two hydroxyl groups in the molecule. One of these is phenolic, while the other is alcoholic. The hydrogen of the former, upon replacement by a methyl group, produces codeine.

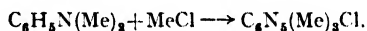
The classical method for methylation is analogous to the well-known Williamson's reaction, viz. by the action of methyl iodide on the sodium salt of the phenol:



The yield is, however, poor and the process has not proved to be economical enough for adoption on a commercial scale. A number of more suitable processes have been worked out of which the most successful method has been found to be the reaction employing trimethyl-phenyl ammonium hydroxide<sup>4</sup>:



The preparation of the methylating agent consists in treating dimethylaniline with methyl chloride in an autoclave:



The chloride is converted into the hydroxide in the presence of alkali. During the last war, the demand for codeine for the Defence Services in India was very urgent and synthetic production had to be attempted. The work was taken up and carried out under the writer at the Control Laboratory of the Central Board of Revenue at New Delhi. A tentative method was worked out and later developed using dimethyl sulphate<sup>5</sup> as the methylating agent following the usual procedure adopted for the manufacture of dionine (ethyl morphine). Codeine as the

phosphate was regularly produced in small quantities and supplied to Defence Services thereafter up to the end of the war. With the cessation of hostilities the demand eased off and the production has been given up. The *Alkaloid Works* at Ghazipur is being fitted up with adequate equipment to adopt the latest process to supplement any demand for codeine beyond what is recovered from opium during the manufacture of morphine. In the meantime, manufacture of codeine from morphine on a small scale is being carried out by use of trimethyl-phenyl ammonium hydroxide (prepared in the laboratory) as the methylating agent<sup>6</sup>.

#### Planning for the Future

A market has to be found abroad for our surplus stocks of opium alkaloids. As mentioned earlier, the world demand for morphine and codeine is about 1,48,000 lb. annually. Out of this India could undertake to produce up to 10,000 lb. now and, if necessary, the forced production could be increased by providing additional units of equipment. The consumption of *Akhari* opium is bound to dwindle progressively, because the Government of India are committed to the policy of prohibition. International opinion also insists upon the prohibition of the use of opium as an intoxicant. It is, therefore, necessary to organize sale of Indian opium alkaloids in overseas markets as early as possible so that increased production of these drugs could be synchronized with the decrease in the production of excise opium. The Government of India are making attempts to secure the overseas markets accordingly. A chemical industry of international importance would emerge out with the establishment of suitable overseas markets for the drugs and would bring in considerable profits to the nation. The profits which may be expected would be apparent from the following figures:

	Price per oz. in India	Price per oz. in U.K.
Morphine	Rs. 20/-	Rs. 33/ 8/-
Morphine hydrochloride	18/-	27/14/-
Codeine	29/-	31/ 2/-

Besides this expected direct profit, the resulting employment at home and prestige abroad are also to be recorded on the credit side. Sale of raw opium to foreign manufacturers is an unsound proposition economically, politically and from the point of international prestige. The Government of India have now restricted the sale of raw opium to foreign countries, and it is likely that such sales would be totally prohibited hereafter.

As an illustration of the attempt that is being made by U.K. manufacturers to capture markets for opium alkaloids, it may be mentioned that the pre-war capacity of U.K. was 2.14 tons of these drugs corresponding to some 20 tons of raw opium. During the year 1946 and 1947, however, U.K. purchases from India amounted to 200 tons nearly.

#### Field for Research

As mentioned earlier, narcotine is a waste product of the opium alkaloid industry. The constitution of this base has now been established. It is a tertiary base containing three methoxy groups. Investigations would be worthwhile undertaking with the idea of manufacturing a chemical or intermediate for use, e.g. in the dye industry or in the manufacture of synthetic drugs.

Another peculiar constituent of opium is meconic acid, which is a cyclic compound containing two carboxyl groups and an alcoholic hydroxyl group in its molecule. Attention of organic research chemists is drawn to this compound which may be made available in considerable quantities. India's want of organic chemicals in general and cyclic compounds in particular should be remembered in this connection.

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# Recent Developments in the Field of Highpolymers

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**M**ACROMOLECULAR substances are equally interesting and important from the scientific and from the practical angle; their behaviour in solution and bulk can in most cases be explained by the existence of a net-work of entangled, long, flexible chain molecules, which attract each other by intermolecular forces and exhibit a certain tendency to form areas of higher geometrical order. The ultimate mechanical, thermal and electrical properties of such polymeric materials depend in a rather complicated manner on the various qualities of the individual macromolecules, and it will be the purpose of this paper to describe briefly our present knowledge of the connections between molecular structure and macroscopic properties of highpolymers. In order to obtain a vivid picture of the present state of development, I shall try to choose a number of actual problems in the field and explain how the application of certain rules has helped to improve the properties of polymers in a desired direction. The materials under consideration cover a wide range of mechanical behaviour, some of them are soft and rubbery, others are soft and plastic, others again are very hard and rigid and others finally are tough and flexible. From the practical point of view, highpolymers are used to make plastics, rubbers, films, fibres, or lacquers, etc., and it may be appropriate to discuss the modern trends in each of these fields and correlate the essential properties of representative materials with the structure of their molecules.

## Trends in Plastic Research

One interesting line of development is, at present, to prepare organic plastics with very high softening points and with a great resistance to chemical changes at high temperatures. The normal organic plastics, polystyrene, polyethylene, polymethylmethacrylate, have softening ranges around 100°C. and start to deteriorate around 400°C. On the other hand, the inorganic polymers in the basis of silicates, borates, etc., which have

softening ranges around and above 300°C., exhibit distinct brittleness at lower temperatures and are stable up to 800° to 1,000°C. It would obviously be very interesting to bridge the gap between the softening range of organic (100°C.) and inorganic (400°C.) glasses and to have at our disposal transparent, colourless, hard but not brittle plastics which possess softening points between 100°C. and 400°C. and are chemically sufficiently stable to undergo reversible softening and hardening in the course of such processes as extruding, spinning, casting, injection moulding, etc. Let us consider the possible approaches to the solution of this problem. It will evidently be reasonable to start with the plastics already known and explore how their properties could be modified in the desired direction. This leads us to the question: why is the softening point of polystyrene so low? The answer is: the molecules are of the hydrocarbon type and, therefore, do not exert exceptionally strong attractive forces on one another. They are also rather flexible and can easily slip along each other under the influence of thermal motion. The *small attractive potential* between the chains and their relatively *high flexibility* are the two reasons for the low softening. As a consequence of this motion we have two possible ways to raise the softening range:

(1) to increase the mutual attraction between the chains; and

(2) to stiffen the individual chain molecules.

Both ways have been taken and in both directions improved products were obtained. To increase the intermolecular attraction between the macromolecules it is necessary to introduce polar groups or groups of high polarizability. As one wants to keep the material hydrophobic and electrically non-polar, one cannot use carboxyl, carbonyl or hydroxyl groups and is restricted essentially to such atoms and groups as the chlorine atom and the nitrile group.

In fact, great success was obtained with such simple styrene derivatives as *p*-chloro-

*p*-cyano-, 2,5-dichloro, 2,5-dicyano and 2,4,6-trichloro styrene. They cover a softening range from 100° to 150°C. and have almost the same excellent electrical properties as polystyrene itself. There exist certain difficulties in reaching sufficiently high degrees of polymerization with these materials and arriving at completely colourless samples; but there is little doubt that these difficulties will be overcome as one obtains monomers of high purity and works out appropriate catalysts and activators. The introduction of polar groups with a high attractive potential can also be effected by copolymerizing styrene with a monomer containing such a group. In this case one does not depart from the cheap and readily available basic monomer completely, but *modifies* it with a comparatively small amount of an auxiliary monomer. In view of water repellency and electrical properties it is again indicated to use the nitrile group or the chlorine atom. In fact, copolymers of styrene with acrylonitrile, fumaronitrile, vinylidene cyanide and pentachlorostyrene have been prepared. Their softening ranges extend from 100° to 200°C.; their electrical, mechanical and optical properties are perfectly acceptable. Equally successful was the other line of approach, namely the synthesis of chains with lesser internal flexibility. Macromolecules of the polystyrene type can be best stiffened by introducing larger side groups which impede the free rotation about the single carbon-carbon bonds in the backbone chain. Polymers of vinylnaphthalene, vinylphenanthrene, vinylfluorene, vinylcarbazole, acenaphthalene and vinyl dibenzofuran have been prepared, and it was, in fact, found that they cover the softening range between 150° and 250°C. and still have excellent optical, mechanical and electrical properties. It is evident that one also can combine the two principles enumerated above and can, at the same time, stiffen the chains and introduce groups of strong intermolecular attraction. Materials such as the copolymer of fumaronitrile and vinylfluorene or as the polyvinyltetrachloronaphthalene and the poly-dichloro-*p* dichlorodiphenyl ethylene are examples for such combinations.

It seems that one can arrive at hard, tough, transparent, colourless plastics which have softening points up to 300°C. and exhibit excellent electrical properties and remarkable chemical stability. In order to illustrate the principles involved, we have

started with polystyrene as original polymer and have introduced chemical changes in the monomer or applied copolymerization to arrive at higher softening plastics. We can, of course, also start with polymethylmethacrylate and introduce analogous modifications. They have, in fact, been carried out in various ways and one has found that the introduction of polar groups in the chloro- and cyano-acrylates, the copolymerization of methacrylates with acrylonitrile, fumaronitrile, etc., and the introduction of bulky groups in phenacrylates, cyclohexyl methacrylates, etc., leads to a whole series of interesting polymers which combine excellent moulding properties with perfect optical clarity, remarkable ageing performance and with softening ranges up to 250°C.

Very interesting materials are obtained if one uses polyethylene as a starting material. Polyvinylchloride, polyvinylidene chloride and polyacrylonitrile are the simplest materials which one obtains if one starts introducing polar groups into the backbone chain of polyethylene. The softening range rises from 100° to about 250°C. and an almost incredible number of useful things can be made from these three polymers in the pure and plasticized state.

Copolymerization of ethylene with polar monomers also leads to higher softening materials: it meets, however, with the difficulty that ethylene, as an almost perfect gas, has to be polymerized and copolymerized under very high pressures which adds to the cost of the final product and makes copolymerization with a liquid monomer somewhat difficult. The third possibility, namely the stiffening of the principal chain by heavy substitution has led to a whole series of extremely interesting new plastics which have softening ranges as high as 350°C. and can be exposed to temperatures up to 400°C. for a prolonged period without decomposing. The first material of this type was the polytetrafluoroethylene which represents a relatively soft, fatty, opaque material of excellent electrical properties, extreme toughness and a softening range near 350°C. It is very stable against any kind of chemical attack but has the disadvantage that it cannot be dissolved and not really be molten. Around 350°C. it sinters to a very viscous, opaque mass which can be brought into certain forms by high pressure. Much better moulding properties are exhibited by the poly-trifluoro-monochloro-ethylene which melts to a transparent mass around 300°C.

and has otherwise almost the same electrical, mechanical and chemical properties as the poly-tetra-fluoro-ethylene. A large number of other heavily substituted ethylenes have been, and are being, studied in connection with the syntheses of high-softening plastics; they all follow the principles outlined above either to distribute groups with strong molecular attraction along the chains or to decrease the internal flexibility of them. Summarizing, it can be said that the application of these working hypotheses has been extremely successful and that, as a consequence, we know today about 50 organic polymers, the softening point of which cover the whole range from 100° to 350°C.

#### **Synthetic Rubbers with Exceptional Qualities**

There exists a few exceedingly interesting problems in the field of synthetic rubbers. One of them is to produce substances which maintain a long-range low-modulus elasticity down to temperatures as low as -80°C. or -100°C. without being too soft at room temperature and above. The typical rubber-like elasticity of polymeric materials is connected with the presence of very long and very flexible chain molecules which are linked to each other by a few strong bonds. To comply with our requirements it is, therefore, necessary to build up an irregular molecular network of chains which have a very high internal flexibility, do not exhibit any large overall attraction in respect to each other but are firmly connected with each other by strong bonds being distributed in a random and infrequent manner over the whole system. A rubber can, in this sense, be considered to be a very fluid liquid with a superimposed system of widely scattered and irregularly arranged fix-points. These strong intermolecular bonds, which do not break during reversible extension and contraction of the samples, are responsible for the reversibility of the deformation. Their infrequent occurrence permits a long-range extension and rapid change in shape. Such fix-point systems can be produced in different ways, the most familiar being the vulcanization or curing of rubber with sulphur or sulphur compounds. One can also establish other strong chemical bonds between the macromolecules of the rubber material with the aid of polyfunctional monomers, di-iso-cyanates, peroxides, etc. It is not necessary that the permanent cross-links of the flexible molecular net-work must be established by primary valence forces;

they can also be caused by very strong adsorption of interfacial attraction.

This is probably the reason for the substantial improvement of the reversibility of contraction if one incorporates into an uncured rubber stock active fillers with large specific surfaces such as carbon black, zinc oxide and the like. Parts of the flexible rubber chains are strongly adsorbed and firmly held at the surfaces of the very small, colloiddally distributed carbon black particles, which assume in this manner the rôle of strong irreversible cross-links. Finally, it is possible to establish strong, if not permanent, bonds between the flexible chains by forcing small crystallized domains in the rubbery material. These minute areas of high lateral order of the macromolecules prevent them from slipping along each other in an irreversible manner and avoid the occurrence of a permanent set, even if the material is kept at a high elongation of a prolonged period. On releasing these crystals melt again and the contracted sample reassumes its soft and easily extensible character. One can also express this situation by saying that uncured rubber builds up its own cross-linking system of fix-points as it is more and more elongated by producing here and there a very small crystallized arc in which the chains are bonded together so firmly that they cannot slip along each other irreversibly and do not permit the development of a permanent set. As this process of crystallization progresses with increasing extension, the material becomes stiff and hard, as most rubbers do in the domain of high elongation and would stay so if the crystalline domains would not melt again as soon as the external forces cease to act. If one wants to have in a synthetic rubber the advantages of such a self-produced, well-controlled fix-point system and, at the same time, requires that the material should be soft and elastic at very low temperatures, it is apparently necessary that the crystals produced during stretching possess a very low melting point.

One structural requirement of a rubber which can be successfully used at very low temperatures will, therefore, be that its macromolecules should have a certain tendency to crystallize but that these crystals should have a melting point or melting range as low as -80° or -100°C. Let us now consider the significance of the fluid part of the structure, namely of the randomly entangled net-work of flexible chains which represent the bulk of the rubber material. This

liquid-like system is responsible for the tendency of a stretched piece of rubber to contract and to return to its original shape. In the normal, unstretched state these molecules represent a mass of randomly coiled and irregularly entangled chains, the individual segments of which can move past each other with a certain ease similar to the way the molecules of a viscous liquid can be displaced by the phenomenon of laminar flow. The higher the viscosity of a liquid, the slower is the displacement of its molecules under a given external force, and hence, the higher the internal viscosity of a rubbery material, the slower it will move if one attempts to deform it. That shows that if we want to get an easily deformable soft rubber, we must keep the internal viscosity as low as possible in order to make all reactions of our material as rapid and instantaneous as possible.

If one subjects a cluster of such randomly entangled, flexible polymer molecules to an external shearing force, their segments will flow as if they were individual molecules of a viscous liquid and, as a consequence, the long chain molecules will be disentangled, straightened out and oriented. This brings them into a state of decreased randomness which, according to the second law of thermodynamics, will have the tendency to go back to the original higher degree of randomness as soon as the external conditions such as temperature, pressure, acting mechanical forces, etc., permit. This is exactly what happens if we relax the stress at the extended rubber sample: the chains try to return to the state of maximum randomness (maximum entropy), coil up in an irregular manner and the sample contracts back into its original unoriented state. The degree with which this return is reversible depends upon whether any permanent displacements have occurred in the system and is connected with the strength of the fix-points which we have discussed above; the rate with which a given rubber material returns into the relaxed state depends upon the viscosity of the mass of irregularly entangled chain segments. If this viscosity is high, it will take a long time until an individual chain segment will diffuse back into its most probable position under the influence of the thermal motion, the average kinetic energy of which one degree of freedom is given by  $kT$ . As a consequence the change in shape of a stretched band of such a material is slow; it creeps back into the original, unstretched shape and will be a slow and

sluggish rubber. On the other hand, if the viscosity of the rubbery liquid is small, the individual segments will move very rapidly and we shall have a quick and snappy rubber. If we want this property to extend to very low temperatures, we must try to avoid that the internal viscosity increases with decreasing temperature, which is particularly important because the retracting tendency decreases with lowering of the temperature, as it is proportional to  $kT$ . This brief and, evidently, only qualitative analysis of our problem permits us to summarize the requirements for a material which remains rubbery even at very low temperatures as follows: we should synthesize long, flexible, chain molecules which have a very small internal viscosity in the randomly entangled (amorphous) state. This viscosity should not increase too much with a lowering of the temperature and should, if possible, still be fairly small at  $-80^{\circ}\text{C}$ . or  $-100^{\circ}\text{C}$ . The molecules should have a good facility to crystallize as soon as they are parallelized by our external force, but the crystals should have a melting point as low as indicated above. If a material of this character is to be used with success at higher temperatures, it will be necessary to superimpose over it a system of permanent chemical cross-bonds. Hence, our molecular chains should contain here and there, at irregular and infrequent points, certain atomic groups which permit the establishment of chemical cross-links. In this way we have now reduced our practical requirements of macroscopic behaviour to certain features of the molecular structure of the desired material and we shall now, as the next step, discuss the chances which exist to arrive actually at substances which combine the above molecular feature. Starting with the most essential requirement of a low internal viscosity and of a small temperature coefficient of it, one is evidently led to substances having a low overall intermolecular attraction, that is to hydrocarbons or fluorocarbons. The presence of polar groups along the chains will certainly be unwelcome, not only because of their strong intermolecular attraction, but also because they would impart to the material a certain degree of swelling in water, which is obviously not desirable in a material for our purpose. At first sight one could be inclined to think that polyethylene would be a suitable polymer for a low temperature rubber, because the material crystallizes easily and has no particularly attractive forces; but it



turns out that the chains of this polymer are so smooth and can be packed so easily in a crystal lattice that the melting point of the crystalline phase is too high. It is true that polyethylene remains soft and flexible even at very low temperatures but it does not exhibit any long-range, reversible elasticity because of the limited flexibility of the normal paraffin chain and of the too easy crystallizability of the material. It appears, therefore, reasonable to consider spacing the individual chain somewhat farther away to make the crystal lattice less stable. It is understood that we should not prevent crystallizability altogether, because we want crystals to be formed during extension in order to get a gradually increasing number of fix-points, but the crystallized domains should be so unstable that they will melt at very low temperatures. If we first consider using methyl groups to space the chains laterally farther apart, we are led to polypropylene. This polymer has received considerable attention, but for some reason it has not yet been possible to synthesize samples of sufficiently high degree of polymerization to produce a rubbery material of high strength. The next material is polyisobutylene which can be polymerized to very high polymerization degrees and is, in fact, an outstanding synthetic rubber with excellent properties at very low temperatures. It crystallizes remarkably easily upon extension but the crystals disappear again upon relaxation at very low temperatures. In order to facilitate vulcanization, isobutylene has been copolymerized with small amounts of isoprene or dimethyl butadiene, introducing widely spaced double bonds along the chains which are capable of reacting with sulphur and produce strong cross-links between the macromolecules. Considering polydienes it would appear that polybutadiene is a hopeful candidate. The conditions, however, are less favourable than one would expect because of the following facts:

(a) If the material is a pure 1,4-addition product, one has possibilities for *cis-trans* isomerization. A continuous *trans* chain is probably the actual configuration of this polymer; it is somewhat similar to polyethylene and forms too stable crystals to be good at low temperatures. If it were possible to synthesize a continuous *cis* chain, it would probably be a polymer which crystallizes easily and at the same time forms crystals having a low melting point. It has not yet been possible to prepare such

a material but its synthesis evidently would offer an interesting chance to arrive at a cheap superior low temperature elastomer. A polymer in which *cis* and *trans* configurations alternate randomly would be a good material from the point of view of general softness, but would probably not crystallize and, therefore, not be an ideal solution of our problem although it might have good properties at low temperatures, it hardly would satisfy expectations at normal and somewhat elevated temperatures because of lack of crystallization.

(b) The introduction of one methyl group in the 2 or 3 position leads to isoprene, the *cis* polymer of which is natural rubber, whereas the *trans* polymer is guttapercha. Looking at the molecular models of these two isomers it becomes evident that the smooth, paraffin-like chains of guttapercha can be much more easily arranged in a crystal lattice than the bulky and kinky chains of rubber. As a consequence the crystals of rubber have a much lower melting point than those of guttapercha. Both materials crystallize upon stretching, but rubber relaxes vigorously and rapidly while guttapercha remains in its stretched, and oriented state up to temperatures of about 70°C. Synthetic polyisoprene is probably a mixture of the two isomeric configurations and has, in fact, properties which are somewhere between rubber and guttapercha. The use of two methyl groups in the 2 and 3 position as spacers has first led to a polymer which was very soft but did not show sufficient strength to be regarded as a good synthetic rubber. However, it secures that the polymerization degree of these materials has not been high enough to permit the development of superior properties. Recent experiments have, in fact, shown that very interesting rubbers can be made from 2,3-dimethylbutadiene alone or in conjunction with other monomers such as isobutylene and styrene if the reaction is carried out under such conditions that a high degree of polymerization is reached. Another interesting case in which methyl groups are used as spacers is that of the poly-di-methyl-siloxane which contains backbone chains of —Si—O— links flanked by methyl groups. It is not easy to produce very long chains of this type but several attempts have shown that if one succeeds in preparing them, the resulting products are rubbery and maintain their reversible elasticity down to very low temperatures.

(c) Chloroprene and fluoroprene can be polymerized to give rubbers with very attractive properties in which the chlorine and the fluorine atoms act as spacers. Both polymers show excellent reversible crystallization upon stretching. Polyfluoroprene maintains its rubberiness down to lower temperatures than polychloroprene and is also more stable against the attack of various chemical reagents. It means also that the ethyl, isopropyl, isobutyl and tertiarybutyl groups can be effectively used as spacers or internal plasticizers in polyvinyl and polybutadiene type compounds such as polyvinyl isobutylether or polyethyl butadiene which show surprisingly good behaviour at low temperatures and are also performing well at elevated temperatures as soon as the polymerization degree is high enough.

It seems, therefore, that the principle of internal plasticizers by spacing of the backbone chains offers interesting leads on how to achieve a decrease of the viscosity of the elastic liquid in a rubber and hence is a good working hypothesis for the synthesis of rubbers which remain soft and elastic even at very low temperatures.

#### New Developments in Fibres & Films

A very important field of practical application of macromolecules is the production of fibres and films, where many problems are still awaiting solution. The most outstanding property of a textile fibre is its ultimate tensile strength. In textile industry this is expressed in gm. per denier (where "denier" measures the cross-section of the filament under consideration). The tensile strengths of natural fibres are given briefly in the following table:

Wool ranges from	0.8-2.2 gm. per denier
Cotton	1.5-4.0 "
Silk	3.5-5.0 "
Hemp, flax,	
ramie	4.0 to 8.0 "

The next important textile property is the elongation to break, which expresses how far a filament can be extended before suffering irreversible damage in form of a break. The protein fibres—wool and silk—have relatively high elongations to break, which range between 20 and 40 per cent, whereas cotton breaks at elongations around 10 to 15 per cent and the stronger cellulose fibres between 5 and 8 per cent; these fibres are strong but stiff. They are useful for many a practical application but leave

many other textile problems unsolved. It has, therefore, been a particularly attractive problem of fibre chemistry to prepare fibres which combine high tenacity with high elongation to break. Before we discuss the possible ways for the solution, let us first consider the conditions in the case of the natural fibres.

(a) Proteins are built up from polypeptide chains which carry many different substitutes and are characterized by the occurrence of a CONH (peptide) group in the backbone chain. These groups start strong intermolecular attraction between the long chains and, in general, favour the existence of crystalline domains. Whenever the substitutes are not too irregular such as in silk and in various synthetic polypeptides, one obtains relatively well-crystallized fibres which have a remarkable strength, but also a high elongation because there remains always a considerable part of the material in the disordered state. For the great internal flexibility of the polypeptide chain these amorphous domains exhibit rubber-like elasticity and are responsible for the relatively large reversible elongation of silk and other natural and synthetic protein fibres which exhibit crystallinity. If the substitutes at the polypeptide chains are of various nature and irregularly distributed, they impede crystallization and prevent the development of high strength. Fibres of this type, such as hair or wool, have, therefore, low tenacities but remarkably high reversible elongations.

Cellulose, on the other hand, consists of long chains of glucose which are intrinsically much stiffer than the polypeptide chains and carry a large number of hydroxyl groups which make strong intermolecular attraction possible. Cellulose fibres, as a consequence, are intrinsically stiffer than protein fibres but can have a very high tensile strength as soon as the chains are arranged so regularly that most of their hydroxyl groups contribute to intermolecular bonding. In such cases, however, the elongations to break become very small. If one wants to combine high tenacity with high elongation to break, it will obviously be necessary to build up long molecular chains of a high degree of internal flexibility which, at the same time, show a considerable tendency for crystallization. Evidently, it is necessary to strike a delicate balance; if the tendency for crystallization becomes too great, not enough of the material will be left

in the amorphous state and the fibres will be very strong but stiff. If, on the other hand, *the flexibility is overwhelming and the intermolecular attraction too weak*, there will result a material with a low stiffening point, which will not give the necessary strength to fibres made out of it. Most recent approaches to synthesize superior textile fibres are of different ways to arrive at a compromise between the flexibility of the backbone chain of synthetic macromolecules and the magnitude of intermolecular attraction between them. Probably the most successful solutions, at present, are the polyamides or nylons. There one maintains the peptide group CONH as the element which provides intermolecular attraction but eliminates the bulky side groups which space the individual chains apart, loosen the structure up and prevent the development of high strength. If one would stick to *d*-amino acids, however, one would arrive at very highly crystalline materials which are infusible, insoluble and give very strong but stiff fibres. Somehow one has to insert the element of flexibility without interfering too much with intermolecular attraction.

In the case of nylon this is done by introducing between the peptide groups along the macromolecules, relatively long, flexible paraffin chains of 6, 8 or 10 carbon atoms. These hydrocarbon segments do not contribute to the intermolecular attraction but they do not space the chains apart and hence do not lessen their lateral cohesion; they only introduce internal flexibility and, hence, facilitate the development of rubberiness in the structure. This leads to fibres which can be crystallized to a considerable extent by cold drawing and as a consequence possess tenacities as high as 8 and even 9 gm. per denier. The forces which bond the macromolecules laterally are essentially hydrogen bridges between the NH groups of one chain and the CO groups of the adjacent chains. They represent a rather strong lateral bonding and are responsible for the high melting point of the various nylons and for the strong resistance against swelling agents and solvents, all properties which are extremely valuable from the textile point of view. There are, however, even after cold drawing, enough disordered areas left to provide for a considerable amount of reversible elongation and for a high elongation to break. The longer the inter-spaced hydrocarbon chain pieces are, the polymers assume a more and more

plastic character and, in the limit, have the characteristic properties of polyethylene. *They are very interesting products as far as injection moulding is concerned*, but are not really outstanding fibre formers. Recently it has been found that high-withstanding and easily crystallizable polyesters can be obtained if one combines ethyleneglycol with aromatic dicarboxylic acids such as terephthalic acid, diphenyl-dicarboxylic acid or naphthalene dicarboxylic acid.

These acids introduce into the chain a certain element of stiffness which facilitates crystallization in spite of the fact that the lateral binding between the chains is not excessively high and makes this special group of polyesters, the so-called terylenes, to be particularly interesting polymers for the production of strong, high-binding fibres, which have somewhat less extensibility than the polyamides but compare favourably with them from the point of view of molecules and price. If one wants to make a fibre of rubber-like character, it will obviously be necessary to maintain a few irregularly spaced, relatively strong lateral bonds between the chains, but space them otherwise sufficiently apart and weaken their average intermolecular attraction so that they will form crystalline domains if parallelized, but crystalline domains of such low stability, that their lateral order collapses again as soon as the stress in the filament is released. One way of achieving this goal is to use a mixture of several dicarboxylic acids, glycols or diamines instead of only one type of each component. This blending produces an irregular arrangement of the points of strong lateral attraction along the length of the macromolecules; at those points, where CO and NH groups of adjacent chains are in favourable position, there will be considerable intermolecular attraction, whereas in between, where the CO and NH groups of neighbouring chains are out of phase, there will be only weak lateral attraction. Fibres of films made out of such polymers show, in fact, typical long-range, rubber-like elasticity. Another way to arrive at the same result is to space the chains apart by bulky, non-polar substances and to reduce the frequency of lateral hydrogen bonds by using alkyl-substituted diamines. It is evident that many combinations are possible to introduce the principles of rubberiness in a polyester, polyamides or polyurethanes and their sorts, in fact, a whole spectrum of polymers of this type ranging from low

melting, tacky rubbers to rigid, strong and high melting plastics and fibres.

If one applies our general principles to the question of fibre-forming polyvinyl derivatives, it becomes evident that the following factors have to be combined: high molecular weight, strong intermolecular attraction, sufficient internal flexibility of the chain, high stability against the action of heat, light and oxygen. Many polyvinyl derivatives have been studied from the point of view of their units as fibre formers and, in fact, relatively good filaments can be obtained from polyvinyl alcohol, polyvinyl chloride and polyvinylidene chloride. It means, however, that a real satisfactory economic compound of all of the above-mentioned qualities have only been obtained by the use of polyacrylonitrile. Fibres spun of solutions of this material are called "orlon" and combine high tenacity, soft handle, good resistance and pleasant lustre in a remarkable manner. They excel also most other textile materials in their resistance against the deteriorating influences of outdoor uses. It is very probable that other polymers, particularly copolymers consisting of two or more vinyl and acryl-type monomers, will perform equally well and, maybe, even better than "orlon" in certain special requirements.

Altogether it can be said that the modern full synthetic fibres are distinctly superior in most qualities to their natural counterparts, the only unsolved problem being at present a synthetic material of wool-like character with high tensile strength and good resistance against atmospheric influences and moulds.

Turning finally to the discussion of fibre-forming polymers, their main requirements are briefly enumerated and discussed. Qualities like clarity, transparency, good resistance against tear and high softening points are evident, but others such as flexibility at low temperatures, heat capacity and resistance against the permeation of water vapour are also of great importance.

Cellulose, in the form of cellophane, is a fairly satisfactory material for many purposes, but its tear strength is relatively poor; it has no heat-sealing capacity and offers no resistance against the diffusion of water vapour. The two last properties can be successfully improved by coating cellophane with a thin layer of a heat-sealing hydrophobic resin, a procedure which leads to an improved type of cellophane. It would, however, be more satisfactory and probably at the end also more economical if all essential properties were combined in one single polymer. As a consequence, many studies are carried out on exploring the fibre-forming properties of such materials as polyethylene, polyacrylonitrile, polyvinylidene chloride, polyesters, polyamides, etc., with the result that there is an almost unlimited number of polymers and copolymers which combine the above-mentioned natural properties of fibre-forming material in many different ways and lead to fibres of very attractive and interesting properties. It would probably be premature to say which of these polymers will finally win out as the best and cheapest solution of the problem, but it is evident that a rather rapid technological development is ahead of us in these fields.

# German Dyestuff Industry

K. VENKATARAMAN

## I — I. G. Farbenindustrie, Höchst

THE I. G. Farbenindustrie has already been reported upon by several British and American teams of workers. The object of the visit by the present team, consisting only of one person, was to make a few observations regarding the plant and processes on which information was not available at the time.

Hochst occupies an area of about 370 acres and normally employed 12,000 workers. The factory manufactured a very wide variety of dyes, intermediates, pharmaceuticals, heavy chemicals, etc. The factory generates its own power using boilers operating at 100 atm.

It was stated that much of the plant at Hochst was manufactured in their own engineering workshops. Hochst had 90 engineers and 2,000 skilled workmen in the engineering section. Rubber lining and plastic lining (the main plastics used being Igelit, polyvinyl chloride) were carried out locally, but enamelling was entrusted to Eisenwerke Kaiserslauten, Kaiserslauten, Palatinum.

The Hochst plant was characterized throughout by good lighting, ventilation, excellent layout and generous spacing for convenient working up and movement of materials, logical arrangement of plant, and availability of spare units enabling the factory to cope with sudden and varying demands.

The products on which reports are appended may be classified as follows:

Intermediates:

(a) Aliphatic

(b) Aromatic

Dyes

Miscellaneous chemicals

### Intermediates

#### (a) ALIPHATIC

*Dimethyl sulphate* — Dimethyl ether was supplied by the I.G. Factory at Leuna in 12-ton tank wagons under 8 atm. pressure. Dimethyl ether was a by-product at Leuna.

*Diethyl sulphate* — This was made from 92 per cent ethanol. The process from ethylene was also employed.

#### *Acetoacetic ester (Ethyl acetoacetate)* —

The process was worked under licence from Alexander Wackerwerke, Munich. The normal capacity was 120 tons a month, but 40 tons a month are now being produced, the production having commenced in the third week of June 1946. The plant was constructed in 1935-36, and part of the equipment was supplied by Wacker.

Acetoacetic ester is prepared in the pharmaceutical section, but over 60 tons per month were used for dyes. The keten method was probably cheaper, but was not used at Hochst since the Wacker process was found to be more convenient to handle.

Detailed processes for diethyl sulphate and benzoyl acetic ester are appended (App. 2, 3).\*

*Chloracetic acid* — The monthly capacity was 300 tons, and the process employed is interesting. A solution of trichlorethylene is reacted at 130°-140°C. with water or aqueous sulphuric acid in a solution of about 50 per cent monochloracetic acid in sulphuric acid of 97-98 per cent, while the monochloracetic acid formed and dissolved in sulphuric acid is continuously run out of the reaction vessel. The hydrogen chloride evolved is used for esterifying methanol. The reaction vessels are of cast iron, jacketed for steam. The product is purified by continuous vacuum distillation.

#### (b) AROMATIC

*Aniline* — The conventional iron reduction process is adopted at Hochst and Uerdingen, and a catalytic reduction process at Ludwigschafen. Dr. Lanz, Head of the Intermediate Section, was of the impression that the catalytic plant at Ludwigschafen sometimes gave trouble, and aniline was often supplied to Ludwigschafen by Hochst. Dr. Lanz did not know the precise difficulties associated with the catalytic plant, but he personally preferred the ordinary iron process. At Uerdingen the iron oxide sludge was converted into pigments, but not at Hochst. Hochst's

\*The appendices given here represent a selection of processes which have not been reported upon by other investigators.

capacity for aniline was 15 tons per day, using 5 kettles of 12 cu. m. There was no special specification for the iron employed, but this was necessary if the iron oxide sludge was to be ultimately employed for pigment manufacture. The iron used at *Hochst* came mainly from the *Opel* automobile plant in the neighbourhood.

*Chlorobenzene* used to be supplied from *Bitterfeld*, where it was made by the ordinary batch process, but a continuous plant making 30 tons per month is now working at *Hochst* and 3 more units are under construction. The separation of chlorobenzene from dichlorobenzene is effected at *Griesheim*.

*p*-Nitraniline was made in 3 cu. m. autoclaves by amination of *p*-nitrochlorobenzene. It was stated that the process was not entirely free from danger, and a continuous process on a pilot-plant scale had been developed just before the war. No documents concerning the subject were available.

*Phenylhydrazine* was made by the sodium bisulphite, zinc and acetic acid reduction process. The hydrochloride was precipitated, filtered on a centrifuge, basified with aqueous caustic soda in presence of xylene, the xylene extract distilled *in vacuo* and the paste finally purified by vacuum distillation in a copper plant in an atmosphere of nitrogen.

*m*-Xylene & *p*-xylene hexachloride (*m*- & *p*-bistrichlormethyl benzene) — Complete side chain chlorination of *m*-xylene and *p*-xylene is carried out in a nickel reaction kettle with a glass ball installed in the cover for an "electric intensive lamp, 300 watts" (APP. 4).

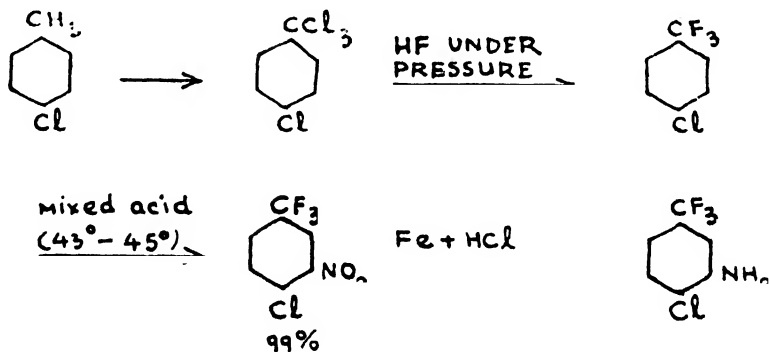
*Terephthaloyl chloride* — The method was to hydrolyse *p*-xylene hexachloride with

water at about 130°C. in a lead-lined steam-jacketed kettle. The iron condenser was lined with bakelite, and hydrogen chloride was absorbed in an Igelit-lined tower.

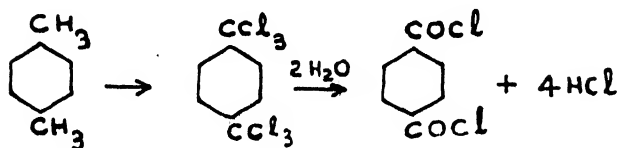
*Benzoyl chloride* was similarly prepared from benzotrichloride.

*Acetoacet-arylates* — The acetoacetic ester-aromatic amine condensation was carried out in xylene solution in an aluminium kettle provided with an aluminium coil and mild steel jacket. The oil jacket was heated by means of a steam jacket. An aluminium column and an aluminium condenser were used for the removal of the ethanol formed in the reaction. The base was dissolved in xylene fed through a pressure filter in an atmosphere of nitrogen. The product was filtered in a horizontal centrifuge in a nitrogen atmosphere, the basket being lead lined and further lined with brass cloth. The arylate was dried in a steam-heated aluminium dryer; an aluminium worm moved the product alternately from one end of the dryer to the other, and when the drying was completed, the material was discharged from one end.

*Fluorine Compounds* — Dr. Scherer, Head of the Southern Section for Intermediates, had carried out considerable research on fluorine compounds as intermediates. Benzo-



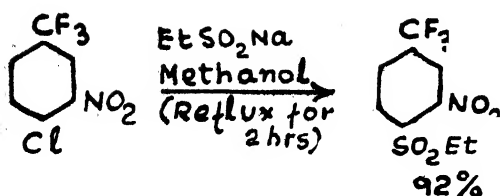
FAST ORANGE RD BASE.



TEREPHTHALOYL bis ACETIC ESTER (APP 3A).

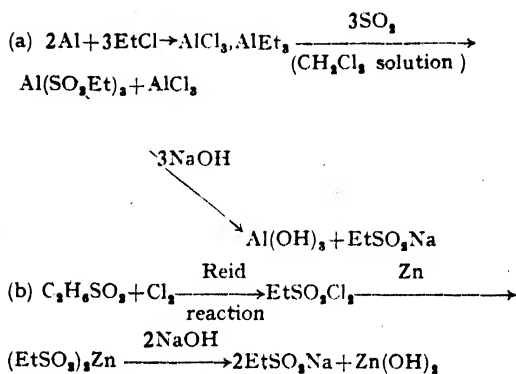
trifluoride was prepared by treatment of benzotrichloride with hydrofluoric acid under pressure, and other compounds containing the trifluoromethyl group were prepared by similar methods. Thus Fast Orange RD Base was prepared by the series of reactions given above.

*Fast Golden Orange GR* was prepared as follows:

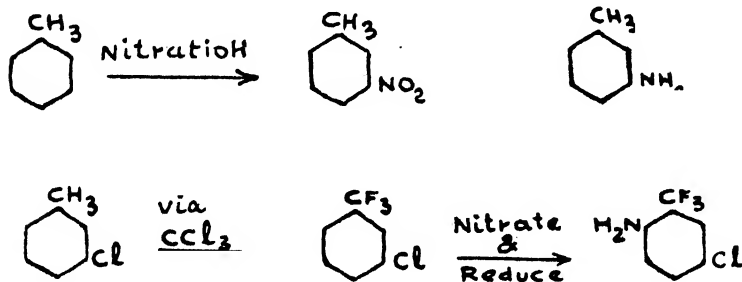


FAST GOLDEN ORANGE GR BASE.

The sodium ethyl sulphinate required in the above reaction was prepared by the following methods:



Fast Scarlet VD Base was prepared by the following series of reactions:



FAST SCARLET VD BASE.

When these bases were employed in azoic dyeing, the influence of the trifluoromethyl group was not merely to improve the clarity of the tone and the light fastness, but to reduce washing fastness. Several Indanthrene dyes containing the trifluoromethyl group were developed before the war, but only one was marketed commercially. This was Indanthrene Blue CLB, which was used for uniforms of the German Air Force, and was prepared as follows.

**Variamine Blue B** — The plant had a capacity of 30 tons per month. The sodium salt

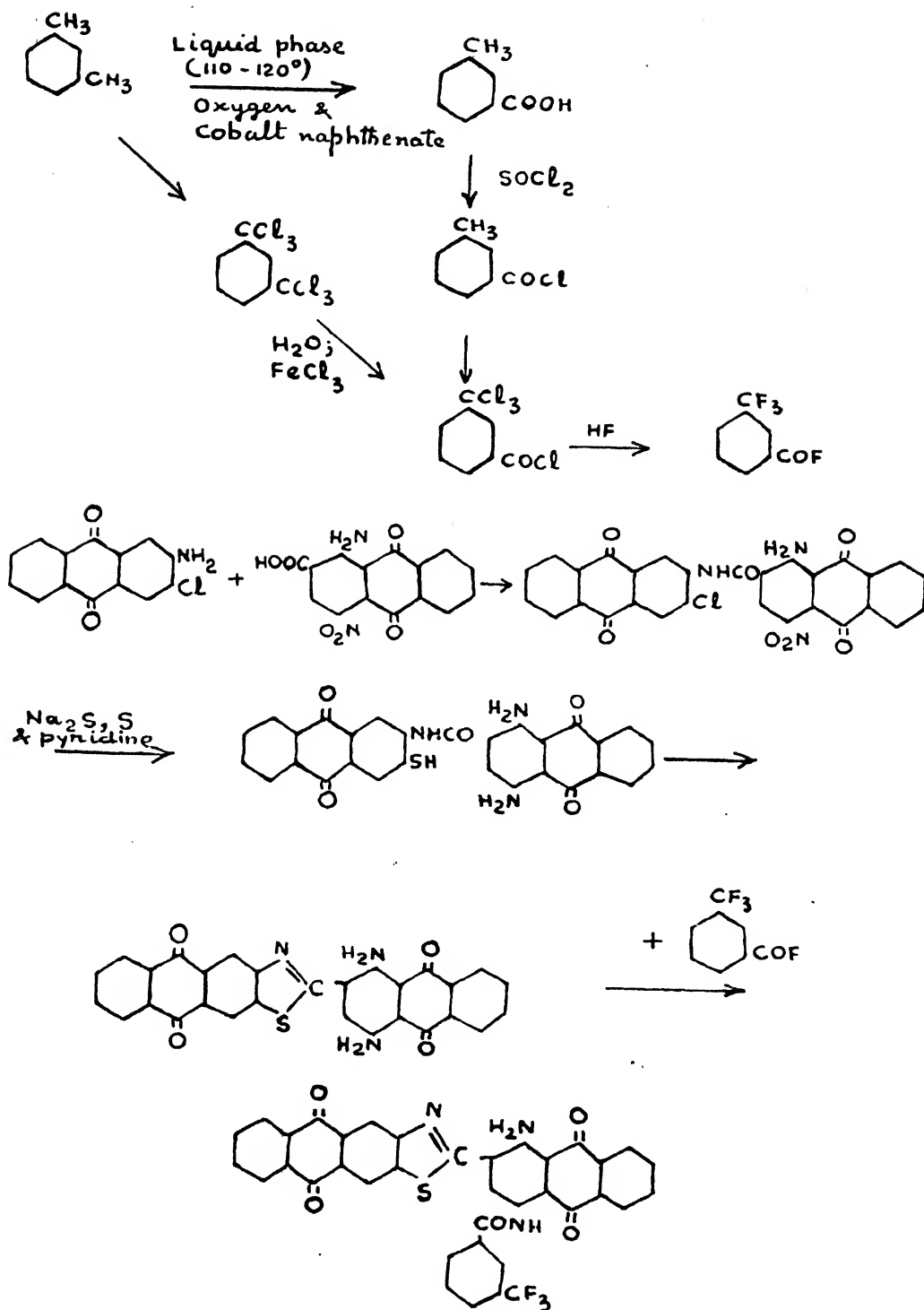
of 4-nitrochlorobenzene-2-sulphonic acid was obtained by salting out the sulphonation mixture. The sodium sulphonate was filtered off on a Nutsch, pasted up with water and heated in an autoclave at 110°-115°C. with

magnesium oxide and *p*-anisidine. The product was precipitated by sulphuric acid, filtered and reduced with iron and acid. The iron oxide was filter-pressed. The amine was salted out and then hydrolysed with 60 per cent sulphuric acid at 115°-123°C. Sulphuric acid was converted into sodium bisulphate by means of sodium sulphate and Variamine Blue filtered off on a rotary filter, and packed directly into drums for despatch to Offenbach for Fast Salt preparation.

Experiments on the preparation of Variamine Blue by condensation of *p*-nitrochlorobenzene with *p*-anisidine under pressure showed that better yields were obtainable by the *p*-nitrochlorobenzene sulphonic acid procedure.

Detailed processes for the following intermediates, fast bases, etc., are appended:

9. Blausulphochloride (*m*-dichlorobenzene-1-sulphochloride.).
10. Blauchtiosaur (2:4-dichlorophenyl-1-thioglycollic acid).
19. *p*'-Methoxyphenyl para-aminic acid (4-amino-4'-methoxydiphenyl-amine-2-sulphonic acid).
22. Amidol acid (1-amino-2-naphthol-4-sulphonic acid).
23. Diazoamidol acid (1-diazo-2-naphthol-4-sulphonic acid).
25. Nitroethylimide base (3-nitro-N-ethylcarbazole).
26. Carbazanil (diaminocarbazyl dichloroquinone).
27. Tetra acid (naphthalene-1:4:5:8-tetracarboxylic acid).
29. Pyrenit S (nitropyrene).
30. Pyren chloride (hexachloropyrene).
31. Perchlorpyrene (tetrachloropyrene tetrachloride).



INDANTHRENE BLUE CLB.



Pyranil crude (dipyrenylaminodichloroquinone).

33. Pyranil pure.

34. Pyram (aminopyrene).

35. Leucopyranil.

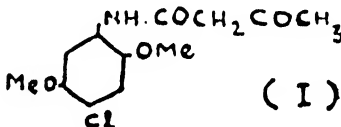
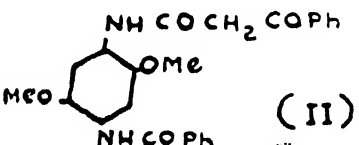
*Alizarine* was manufactured at *Hochst* 15 years ago, but the plant was later used for other purposes.

*As Dyes*—The azo shed at *Hochst*, which is modern and very well laid out, has been reported upon by a British team. There was no provision for the gravity flow of materials in the manufacture of *disazo* and *trisazo* dyes. Diazotization was carried out in wooden vats, and diazo solutions dropped into rubber-lined iron vessels from which the solutions could be blown out into a filter press. The coupling was carried out in wooden vats provided with rubber-lined iron agitators.

Pipe lines for the diazo solution were iron, rubber lined. For glacial acetic acid aluminium pipe lines were employed. The dyes were fed into the filter press by means of bronze pumps. "Paper cloth" was found to be suitable for the filter press. Pigments as well as dyes were filtered through filter presses.

Ice from the top floor was disintegrated in a simple type of disintegrator provided with spike rollers and dropped down chutes into rubber-tired hand carts of aluminium, from which they were discharged as required into vats directly or through chutes.

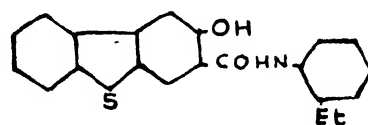
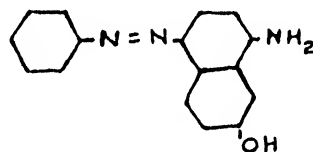
*Rapid Fast Colours*—The composition of various Rapid Fast colours was stated to be as follows; the base of which the anti-diazotate is used, and the Naphtol, are both indicated in the following table:

<i>Rapid Fast</i>		<i>Base</i>	<i>Naphtol</i>
Yellow	GGH GH	Red RC Scarlet GG	AS-G AS-G
	13 GH	Red TR	 <p>( I )</p>
Golden Yellow	IGH	Red RC	 <p>( II )</p>
Orange	GH IGH IRH RG RH	<i>m</i> -chloroaniline 4-chloro-3-amino-trifluoromethylbenzene do <i>o</i> -nitraniline Scarlet GG	AS-D AS AS-OL AS AS-PH ( 2-hydroxy-3-naphthoyl- <i>o</i> -phenetidine
Gold Orange	IGG	2-amino-4-trifluoromethyl-phenyl ethyl sulphone	AS-D
Scarlet	ILH RH	Scarlet GG Red KB	AS-OL AS-PH
Red	B BB FGH GL G2H 13GL ILB IRH RH	Red B Red B Red KB Red GL Scarlet GG Scarlet 3GL Red RL Red TR Red RC	AS AS-D AS-D AS AS AS AS-RL AS-LT AS-OL

<i>Rapid Fast</i>		<i>Base</i>	<i>Naphtol</i>
Bordeaux	IB	Red B	AS-BOX ( undissolved AS-BO )
	RH	Red RC	AS-BS
Blue	B	Dianisidine	AS
Brown	B	Mixture of dianisidine and <i>p</i> -nitraniline	AS
	GGH	Scarlet GG	AS-LB
	IBH	Red RC	AS-LB
	IRH	Scarlet GG	AS-BG
Olive Brown	IGH	<i>m</i> -chloraniline	AS-LB

<i>Rapidogen</i>		<i>Base</i>	<i>Amino acid used for stabilization</i>	<i>Naphtol</i>
Yellow	G	Red KB	2-amino-4-sulphobenzoic acid	AS-G
	GS	Red KB	Sarcosine	AS-G
	GG	Red RC	do	AS-G
	14G	Red TR	Methyl taurine	(I)
	PT		Diazoamino compound from <i>p</i> -toluidine	AS-G
Gold Yellow	IFC	Red RC	Sarcosine	(II)
	IGL	Red KB	do	AS-L <sub>4</sub> G
	R	Red KB	do	AS-G + AS-PH
Orange	G	Orange GC	2-amino-4-sulphobenzoic acid	AS-D
	IGN	GCD	2-ethylamino-5-sulphobenzoic acid	AS-OL
	IRR	RD	do	AS-OL
	R	Scarlet GG	do	AS-PH
Scarlet	IL	Scarlet GG	do	AS-OL
	R	Red KB	2-amino-4-sulphobenzoic acid	AS-PH
	RS	KB	Sarcosine	AS-PH
Red	GS	KB	do	AS-D
	G	KB	2-amino-4-sulphobenzoic acid	AS-D
	IGG	<i>o</i> -Toluidine-4- N-dimethyl sulphonamide	2-methylamino-5-sulphobenzoic acid	AS-LC
	ITR	Red ITR	2-ethylamino-5-sulphobenzoic acid	AS-ITR
	R	Red RC	Sarcosine	AS-OL
Bordeaux	IB	Red B	2-methylamino-5-sulphobenzoic acid	AS-BO
	RN	RL	2-ethylamino-5-sulphobenzoic acid	AS-D
Corinth	IB	Corinth LB	2-methylamino-4-sulphobenzoic acid	AS-LT
Violet	B	Violet B	Sarcosine	AS
Red Violet	RR	2-nitro-5- amino-4-me- thoxy toluene	2-ethylamino-5-sulphobenzoic acid	AS-BO
Blue	B	Blue BB	50% sarcosine, 50% methyl taurine	AS
	BN	BB	do	AS
Marine Blue	IB	Variamine Blue B	Sarcosine	AS
	D	Mixture of Blue Salts	do	AS-D
Blue	R	RR Base	Methyl taurine	AS
Marine Blue	B	Blue B	do	AS
do	R	Blue RR	do	AS

<i>Rapidogen</i>		<i>Base</i>		<i>Amino acid used for stabilization</i>	<i>Naphthol</i>
		---	BB	50% sarcosine, 50% methyl taurine	AS-BO
Green	B			Sarcosine, N-methyl taurine	AS-CR
Brown	GN	Azored			
		Salt	KBS		
		Azoblu			
		Salt	RR		AS+AS-G
	IB	Red	RC	Sarcosine	AS-LB
	IBR	<i>p</i> -toluidine		do	AS-LB
	IRRN	Red	RL	2-amino-4-sulphobenzoic acid	AS-LB
Olive Brown	IG	Orange	GG	do	AS-LB
Black Brown	ITR	Red	LTR	2-ethylamino-5-sulphobenzoic acid	AS-BT
	T	Red	RC	Sarcosine	AS-BT
	IT	Azored			
		Salt	RC		AS-BT+
					AS-ITR
Black	IT	Red	B	2-methylamino-5-sulphobenzoic acid	AS-SR+
					$\beta$ -tetralol
					carboxylic
					acid- $\alpha$ -
					naphthyl-
					amide
	MG	Azoblu			
		Salt	MG		
		and	BD		
		(Blue B			
		and	BB)		
	IG	Red	RL	2-ethylamino-5-sulphobenzoic acid	AS-SR+



**Indigo White**—Indigo production at *Hochst* was 400 tons per month of which 50 per cent was sold as Indigo White.

The process for Indigo White was by nickel reduction of indigo. The catalyst was prepared by reduction of nickel carbonate mounted on Fuller's earth.

Indigo reduction was carried out in a horizontal boiler, jacketed for steam, using 20 per cent indigo paste, caustic soda, the catalyst and hydrogen. The leuco solution was siphoned off from the catalyst, and the procedure then varied according as whether the product was to be marketed in the form of paste or grains. For paste the leuco compound was precipitated from solution by

carbon dioxide and filtered off in a filter press using jute cloth. It was then mixed with molasses and evaporated in a Frederking at 50°C. *in vacuo*. This was necessary, since the viscous paste had to be agitated with a powerful 6" diameter scraper agitator. The product was thus evaporated to 50 per cent Indigo White content and dried finally in a vacuum tray dryer.

For grains the leuco solution containing about 17 per cent of Indigo White was mixed with molasses, concentrated in a Calandria ("Wetkowitz heat exchanger"), and dried in trays in vacuum. The addition of molasses was adjusted so as to obtain grains of 50-60-70-80 per cent indigo content.

### Indanthrenes

While detailed processes for a few Indanthrenes are appended, a few general observations are made. Aluminium chloride was stored and handled in lead-lined drums. For reactions involving aluminium chloride and sodium chloride mixtures vigorous agitation was necessary, and a propeller agitator was employed. The reaction was carried out in cast iron enamelled vessels, and the hydrogen chloride evolved was removed by using steam ejector suction. Hydrochloric acid solutions were handled in brick-lined vats and stone Nutsches. For moist hydrochloric acid vapours, pipes were of polyvinyl plastic. Iron pipes were adequate for gaseous chlorine, but where iron contamination was involved, lead pipes were used. In preparing chlorinated Indanthrenes the chloride used was checked up by merely weighing the chlorine cylinders, and no rotameters were employed. In a few cases a modified Pitot tube was used. For chlorinations on a comparatively small scale chlorine in cylinders was used, but for bulk chlorinations (all these chlorinations being carried out in the same shed) such as chlorinated paraffins and chloranil, where more than 100 kg. per hour of chlorine were necessary, the chlorine was led directly from the neighbouring electrolyser house.

Detailed processes for the following Indanthrenes are appended:

41. Brilliant Orange GR and Bordeaux RR.
42. Brown GR.
43. Turquoise Blue GK Powder.
50. Printing Brown B.
51. Printing Brown 3R.

### Indigosols (Anthrasols)

The British method for preparing solubilized vat colours was employed, but it was claimed that the process has been shortened and improved. Recovered pyridine was dehydrated by azeotropic distillation with benzene and fractionated in a suitable column. Iron was used for reduction in most cases — iron from the needle industry for Blue BC and electrolytic iron in other cases. Copper was used only in one or two cases such as Anthrasol Red IFBB.

Detailed processes for the following Indigo-sols are appended:

- 61a. Anthrasol Brown IRRD.
- 61b. Anthrasol Brilliant Pink I<sub>3</sub>B.
- 61c. Anthrasol Red IFBB.

**Miscellaneous Chemicals** — A point of interest in the preparation of heavy chemicals

at *Hochst* was the use of mercury cells for caustic soda and chlorine, of which a detailed account is given in *CIOS XXVI-II*.

Pure sodium sulphide was prepared by an elegant process in which sodium-mercury amalgam flowing out of the cells was treated with the calculated amount of sodium tetrasulphide, leading to pure sodium sulphide.

**Tetranitrocarbazole** — This substance is claimed to be a valuable insecticide for specific purposes. Nitration of carbazole is carried out in the usual manner, but a safety device is used for stopping the flow of nitric acid and blowing a siren in the event of the failure of the agitator. The temperature during nitration is electrically recorded.

For immersing the nitration product containing sulphuric acid, the vessel is of mild steel, lined with polyvinyl chloride and then with bricks. Cooling coils are of lead, the coils being lined with Igelit above the level of the liquor; this is because large quantities of nitrous fumes are evolved and corrode the lead. The thermometer pocket and the agitator are lead lined and given a top lining of Igelit. The product is filtered on closed stone Nutsches. The piping is again lead lined with Igelit. The Nutsch capacity not being adequate, part of the filtration is carried out by a filter press using polyvinyl chloride fabric. For separating any liquid carried over by fumes, an Igelit cyclone separator is provided. The mother liquor, the sulphuric acid content of which is too impure to justify recovery, is sucked off through a pipe and barometric leg straight into the drain. The Nutsch cake is thixotropic and is snuffed up with a little water into a kettle. The paste is dried at a temperature of about 160°C. on a drum dryer provided with brass knives. The cast-iron drums are built for 15 atm. pressure and steam is supplied at 6.5 atm. The dry product is fed into barrels by means of a worm conveyer. (On one occasion, on account of the conveyer outlet choking up, there was a rise in temperature to above 200°C. resulting in a fire.) The vapours from the dryer are washed with hot water to remove tetranitrocarbazole dust, and the water is used for pasting up the next lot of Nutsch cake. The nitrous fumes are finally absorbed in an Igelit column packed with Raschig rings, and provided with an Igelit suction fan.

The tetranitrocarbazole thus prepared has m.p. 284°C., and is marketed in this form. If a pure sample is desired, the crude product (10 gm.) may be dissolved in water (3 l.) at

100°C.; on the addition of N. sodium hydroxide (65 c.c.) the substance dissolves. Sodium bicarbonate (10 gm.) in water is now added and the precipitate filtered off. The red product now melts at 295°C. A yellow product is obtained by using ammonium chloride in place of sodium bicarbonate or by recrystallizing the red compound from dioxane.

A detailed process for tetranitrocarbazole is appended (APP. 62).

In spite of many attempts a practicable method for the synthesis of carbazole had apparently not been developed.

The methods for the estimation of the purity of carbazole isolated from coal-tar being unsatisfactory, a new method was devised. The principle of the method, a detailed account of which is appended (APP. 63) (Ref. *I.G. Analyse* No. 400), is to separate carbazole as N-nitrosocarbazole, which is decomposed on heating with ferrous sulphate solution, and the nitrous gases evolved are measured. Phenanthrene, anthracene and acridine do not interfere.

*Diffuorodichloromethane (Frigen)* — A detailed process for Frigen (Freon) is appended (APP. 64).

*Octadecyl isocyanate* — A detailed process for this intermediate used in the preparation of Persistol VS, which is a useful finishing agent for textiles for the production of water-repellent and waterproofed effects, is enclosed (APP. 65).

## II. — I. G. Farbenindustrie, Ludwigschafen

The object of the visit was to inspect the plant for *o*-benzoyl benzoic acid and its derivatives,  $\beta$ -aminoanthraquinone and Indanthrene Blue R, and to obtain information regarding a few intermediates and dyes.

Dr. Pflaumer, Director of the Works, mentioned that the entire factory had already been surveyed by several British and American teams, and one of the British teams, which stayed for four weeks, collected complete data on all vat dyes. A complete set of photographic copies of all the processes for intermediates and dyes employed at *Ludwigschafen* had been supplied to the American F.I.A.T.

*m*-Hydroxydiphenylamine — This is an intermediate in the preparation of Variamine Blue FG. A mixture of resorcinol (1,550 parts), aniline (1,850 parts) and sulphanilic acid (16 parts) is heated at 185°-190°C. for 45 hr., the temperature then being raised to 230°C. in 10 hr. Sulphanilic acid has now

taken the place of iodine which was originally used and which was not available during the war. The product is fractionated, *m*-hydroxydiphenylamine coming over at 220°-265°C. at 7 mm. pressure. The yield is 2,000 kg.

*BOB Plant* — The plant for condensing phthalic anhydride with benzene, chlorobenzene, etc., and cyclizing the resulting *o*-benzoylbenzoic acids is of a very modern design. A series of four 5 cu. m. vessels and one 3 cu. m. vessel are provided, and the same type of kettle is used for sulphonation of anthraquinone, the preparation of chloro- and methyl benzoylbenzoic acid, and the preparation of butyl phenol. The special features of the plant are large porcelain pipes, glass water-sprinklers and a continuous method for decomposing the Friedel-Crafts reaction product with water and for subsequent stages. For separation of aqueous and non-aqueous layers, horizontal separators are preferred, clearer separation being obtainable from vertical separators.

The aluminium chloride used in the plant is also made at *Ludwigschafen*.

The benzoylbenzoic acids are filtered on rotary filters and dried in a Buetner continuous dryer consisting essentially of revolving plates with large perforations, through which the material slowly travels down the series of plates until ultimately discharged.

$\beta$ -aminoanthraquinone is made from  $\beta$ -chloroanthraquinone in an autoclave heated by internal steam coils (30 atm. steam pressure). Arsenic pentoxide is used in the amination. The product is blown over and filtered in a closed Nutsch, and dried on a Venuleth dryer.

2-amino-3-chloroanthraquinone is prepared by partial amination of 2:3-dichloroanthraquinone.

2:6-diaminoanthraquinone is made from 2:6-disulphonic acid, 25 per cent ammonia and arsenic pentoxide at a temperature of 200°C. and a pressure of 45 atm.

1:4-diaminoanthraquinone is made from quinazarin.

*Indanthrene Blue RSN* — Nickel-iron (0.5 per cent nickel) kettles are used for fusion. There is a screw conveyer for the introduction of  $\beta$ -aminoanthraquinone and an automatic arrangement for the addition of sodium nitrate. A mixture of caustic soda and caustic potash in molten form is fed in. For filtering the leuco compound, a closed-type imperial rotary filter is employed. The leuco compound is then mechanically

fed in through a worm into the oxidation vessel.

**Chlorinated Indanthrenes** — These are made in iron reaction kettles provided with Hoesch stirrers with a speed of 700-1,000 r.p.m. The progress of the chlorination is followed by loss in weight of the chlorine cylinders and by estimating chlorine in samples of the product.

**Indanthrene Yellow G** is prepared from  $\beta$ -aminoanthraquinone in 3 stages. In the first stage, 1-chloro-2-aminoanthraquinone is heated with phthalic anhydride in presence of ferric chloride to produce 1-chloro-2-phthalimidoanthraquinone. In the second stage, 2 mols of 1-chloro-2-phthalimidoanthraquinone are heated with copper powder to produce 2:2'-diphthalimido-1:1'-dianthraquinonyl. After removing the copper by means of sodium chlorate and hydrochloric acid, hydrolysis and cyclization are effected by heating with caustic soda solution.

**Indanthrene Brilliant Green B** — 4:4'-dibenzanthronyl is prepared by heating benzanthrone with caustic potash and sodium acetate in isobutanol solution. Oxidation of the dibenzanthronyl to 16:17-dihydroxydibenzanthrone is effected by means of manganese dioxide and sulphuric acid, and the final stage of methylation by methyl benzenesulphonate.

**Violanthrone (Indanthrene Dark Blue BO)** — In the fusion of benzanthrone with caustic alkali and sodium acetate, carbazole-free anthracene residue is used as flux.

**Indanthrene Direct Black RB** is prepared by the condensation of 1 mol of tetrabromopyranthrone with 2 mols of amino-violanthrone and 2 mols of  $\alpha$ -aminoanthraquinone.

Detailed processes for the following are appended:

67.  $\beta$ -aminoanthraquinone.
68. 6-amino-1:9-pyrimidanthrone (Pyrimidin AFS).
69. Indanthrene Yellow 7GK.
72. Nitrocarbonsaure (1-nitroanthraquinone-2-carboxylic acid).
74. Indanthrene Blue RS.
- 74a. Indanthrene Blue BC.
- 75a. 4, 4'-dibenzanthronyl.
- 75b. 2, 2'-dihydroxydibenzanthrone.
- 75c. Indanthrene Brilliant Green FFB.
76. Benzanthrone-sulfid.
77. Isothrene.
79. Dichinyl C.
82. Indanthren Red RK.
83. Indanthren Olive Green B.

### III. — I. G. Farbenindustrie, Offenbach

The object of the visit was to inspect the plant for  $\beta$ -hydroxynaphthoic acid (BON acid).

The plant is very old and, except the carbonators for BON acid, nothing of special interest was noticed. The only hydroxycarboxylic acids made at Offenbach were BON, salicylic (on occasions) and *p*-hydroxybenzoic acid. The other acids used in the preparation of Naphthols came from *Leverkusen* and other I.G. plants. Yellow-producing Naphthols (G, LG, L<sub>3</sub>G, L<sub>4</sub>G) were not made at Offenbach.

The BON acid plant, though fabricated over 20 years ago, proved to be of great interest. The carbonators, of which there are 6, are heated by cast-in steam coils using steam at 25 atm. pressure, and are provided with powerful heavy duty agitators (1.5 r.p.m.). On account of the great resistance offered by the material to be stirred the agitators were liable to break down from time to time, and there was provision for immediate stoppage of the agitator by means of an electrical resistance indicator connected to the motor driving the stirrer. A feature of the process was that  $\beta$ -naphthol was continuously distilled off during carbonation. The process was stated to yield excellent results, but the plant was costly to construct and maintain.

2 small boilers (2 tons of steam per hour at 60 atm.) were reserved for process work, as the BON acid plant was a complete self-contained unit which included the production of carbon dioxide.

**Stabilized Diazonium Salts** — The drying procedure appeared to be unsatisfactory and the equipment crude in design. Tray dryers operated at 50°C., and provided with safety flaps in case of explosion, were employed. During grinding of Fast Salts with Glauber salt, aluminium sulphate, etc., dry ice was added. The initial size reduction of Fast Salts was carried out in disintegrators made of phosphor bronze to minimize the danger of self-ignition and explosion. After dilution with dry inorganic salts, Fast Salts could be ground in iron mixers consisting of revolving drums, provided with scraper blades working in opposite directions. Each mixer was 1 to 3 tons in capacity. The plant was provided with a very good dust-extracting system, the dust entering a tower sprayed with water before being discharged into the atmosphere.

For making Rapid Fast printing colours, special mixers with flat top, flush with the

floor level, and provided with spiral agitators were employed.

Detailed processes for the following are appended :

91. Nitrazol CF extra.
95. Echtschwarzsatz G (Fast Block Salt G).
96. Naphtol AS-SW.
103. Naphtol AS-LC.

#### IV.—I. G. Farbenindustrie, Mainkur

The main object was to see the Hydron Blue plant.

*Hydron Blue* — The plant was constructed in 1937. With a production capacity of 1,000 tons a year, 714 tons were actually produced in 1937.

*p*-Nitrosophenol — This was prepared in the known manner. A lead-lined horizontal centrifuge was employed for filtration; the basket was lined with copper fabric fastened with copper screws. *p*-Nitrosophenol was safe to handle in paste form, but was inflammable when dried.

For acidification at 0°C. a brick-lined vessel with wooden agitator was used. The solution was introduced in the sulphuric acid by means of a short pipe.

*Carbazole-indophenol* — The critical factor was the temperature. Cooling was effected by calcium chloride solution (d. 1.3) from an ammonia plant with a capacity of 60,000 k. cal. per hour at -40°C., supplied by *Gesellschaft f. Lindle's Eismaschinen, A.G., Hildastr., 6, Wiesbaden*. The indophenol was filtered in a wooden filter press using nitrated cloth or polyvinyl cloth.

The best tests for the quality of carbazole-indophenol were solubility in acetone and thionation. For dissolving carbazole and *p*-nitrosophenol and for the condensation of the two to form carbazole-indophenol, 6 reaction vessels are available of which 2 are provided with jacket and coils for cooling, while 4 have cooling coils only. One of the reaction vessels, reserved for condensation, has 4 banks of cooling coils reaching to the top of the vessel. The others have 3 banks of coils in the lower half of the vessel. Agitation is effected by means of propeller stirrers.

For the reduction of the indophenol, lead-lined iron kettles with wooden agitators, chutes for the addition of ice and large vent pipes are available.

The indophenol is washed in brick-lined vats and filtered on 2 filter presses.

Thionation is effected in butanol at a temperature of 108°-110°C. The iron thionation pot is steam heated and provided with steam pipes for quenching fires and for a little pressure for the discharging pump.

The solvent is distilled off after thionation, steam entering through 2 pipes, one inside the liquid, and the other touching the surface so as to break the foam. The distillation pot is also steam jacketed.

The drying of the dye is carried out in a circular chamber with glazed tiles provided with a disc spray dryer revolving at 7,000 r.p.m. A scraper plough working on the floor of the chamber discharges the material. The hot air outlet passes through filter bags to catch any dyestuff dust. The dryer is capable of evaporating 250 l. of water per hour, and yields 50-60 kg. of the dyestuff.

*Suprafix dyes* for printing are manufactured at *Mainkur*. They consist essentially of mixtures of a vat dye (sometimes as a leuco compound) and of Solution Salt B (sodium salt of N-benzyl sulphonic acid); a booster (such as anthraflavin or ferric sulphate) is also added, groups of dyes requiring specific boosters. The whole is then diluted with glycerin and water as required and colloidalized in a suitable mill. A typical procedure was to mix in a lead-lined vessel 30 per cent glycerin, 10 per cent Solution Salt B, 2.5 per cent ferric sulphate, 10 per cent tragacanth solution (6.5 per cent), 0.3 per cent Preventol and 10 per cent leuco compound of Indanthrene Pink R (or Algal Orange RF) made up with water. The mixture is then worked in a colloid mill and sieved.

Detailed processes for the following are appended :

106. *p*-Nitrosophenol.
107. R-base.
114. Ethyl carbazole.
126. Nitrocarbazole.
129. Anthronaphthylen.
133. Dinasaure (1:1'-dinaphthyl-8:8'-dicarboxylic acid).
137. Pyrazolgelbkali.
138. Indanthrene Yellow GF.
140. Indanthrene Scarlet B.
142. Kuppenbraun IVD (Vat Brown IVD).
147. Kupenscharlach RM (Vat Scarlet RM).
148. Indanthrene Brilliant Orange RK.
153. Sirius Light Yellow RT.
154. Diethyl Red.

**V — Kalle & Co., Biebrich-Wiesbaden**

**Cellulose Glycollic Acid** — Cellulose (wood pulp; 38 per cent cellulose, 6 per cent moisture) (1,000 kg.) in the form of sheets is treated with 18 per cent caustic soda solution in a rectangular iron box between perforated plates at room temperature for  $1\frac{1}{2}$  hr., and submitted (hydraulic press) to horizontal pressure for 1 hr. The weight of the sheets is 2,600 kg. The cage containing the sheets is then lifted and the sheets are dropped into a shredder. The alkali-cellulose consists of 14 per cent sodium hydroxide, 1 per cent sodium carbonate and 32-34 per cent cellulose, and is transferred by means of a worm into a Werner-Pfleiderer shredder mill. The shredded soda cellulose is blown by compressed air through pipes into 2 larger boxes from which it is discharged into wagons, then weighed and dropped into a Werner-Pfleiderer kneading machine together with sodium chloracetate. Each charge in the shredder consists of alkali-cellulose, corresponding to 500 kg. of cellulose, together with 375 kg. of sodium chloracetate. The temperature rises during the reaction and after 2 hr. the material is discharged into a box and therefrom into a rotary dryer, where it is worked for 4 hr. This crude product is Tylose HBR containing 33 per cent sodium cellulose glycolate, 33 per cent water. The material now passes down into a box on to a belt conveyer and is fed into a disintegrator. After passage through a cyclone separator to remove air, and through pipes, where it is blown by compressed air into a higher floor from which it drops through a hopper, the Tylose is finally weighed, and packed in paper bags which move forward mechanically.

**Methyl Cellulose** — Soda cellulose is prepared in the same type of plant as for cellulose glycollic acid. Soda cellulose is then either shredded, or the sheets are placed on a wire and rolled on a wheel into a package consisting alternately of cellulose sheets and wire.

Methyl chloride from tank wagons is pressed into a feeder autoclave where it is heated by steam coils and fed into a reaction autoclave by means of a centrifugal pump. The iron autoclave is nickel-lined, and the pump and pipes are of iron-nickel alloy. The charge per autoclave corresponds to 600 kg. cellulose and 550 kg. of methyl chloride, a large excess of methyl chloride being necessary on account of the formation of methanol of which 16-18 kg. are recovered per 100 kg.

of methyl cellulose. The autoclave is heated externally by steam first to 70°C. and then to 90°-100°C., the pressure being 5 atm. The reaction is carried out for 5-6 hr., the total cycle in the autoclave from loading to unloading taking 8 hr., so that each autoclave could handle 3 batches a day. Methyl chloride is circulated through the alkali cellulose, methanol is condensed, and the residual methyl chloride re-heated and circulated. Methyl cellulose is purified by agitation with hot water and filtration on a horizontal centrifuge. The material is dried in a rotary dryer. When the soda cellulose is used in the form of sheets mounted on wheels, a different type of autoclave is used.

**APPENDIX 2****DIETHYL SULPHATE** (made from ethyl sulphuric acid)

**Principal Uses** — Ethylating agent for pharmaceuticals.

**Equipment** — (1) 1 enamelled vessel, 3 cu. m., with jacket and enamelled stirrer; (2) 2 aluminium receivers, 1 cu. m. each, with a 5-stage steam jet pump; (3) 1 aluminium stock vessel, 4 cu. m.

**Process: Diethyl sulphate** — In (1), 1,100 kg. of ethyl sulphuric acid and 1,950 kg. of dry sodium sulphate are mixed and distilled into (2) within 12-15 hr. under a reduced pressure of 5 mm. Hg. The distillate is stored in (3). Yield: 580 kg.

**Process Control Tests** — Determination of the sp. gr. and strength of ethyl sulphuric acid and diethyl sulphate.

**Raw Materials, Intermediates & Finished Product Specifications** — Sp. gr. of ethyl sulphuric acid: 1.41 at 15°C.; sp. gr. of diethyl sulphate: 1.18 at 15°C.; content: 99.8 per cent of diethyl sulphate and 0.1 per cent of ethyl sulphuric acid.

**DIETHYL SULPHATE** (prepared from ethylene and sulphuric acid monohydrate)

**Equipment** — (1) 1 cast-steel autoclave of 1,000 l. with an admissible maximum pressure of 50 atm. gauge pressure; (2) 1 iron measuring vessel of 1,000 l.; (3) 1 iron measuring vessel of 1,000 l.; (4) 1 iron vacuum distilling vessel of 2.5 cu. m. provided with jacket heating; (5) 1 5-stage steam jet water pump; (6) 2 aluminium receivers each of 1 cu. m.; and (7) 1 aluminium stock vessel.

**Process** — 527 kg. of sulphuric acid monohydrate (representing 307 l.) are filled into the vessel and 328 kg. of ethylene from a steel



bottle are pressed in. The vessel is then heated to about 70°-75°C. by means of warm water, the ethylene being absorbed rapidly. After about 12-16 hr. the main quantity of ethylene is consumed. The remainder is absorbed with difficulty. After about 20 hr. the pressure (5 atm.) is released. When the product has assumed the right density and acidity, the contents of the autoclave are pressed into another vessel. From here the reaction mass is brought in 2 equal portions into a vessel which contains 140-150 kg. of dry sodium sulphate and is distilled at 5-8 mm. reduced pressure in the course of 12-15 hr. The yield amounts to 630 kg.

*Process Control Tests* — The strength of diethyl sulphate is determined by saponifying it with a measured quantity of hydrochloric acid.

*Raw Materials, Intermediates & Finished Product Specifications: Contents of the Autoclave* — The sp. gr. at 20°C. amounts to 1.22-1.25; acidity: 10 c.c. of it correspond to 28-35 c.c. of N. sodium hydroxide.

*Diethyl sulphate* — The sp. gr. at 20°C. amounts to 1.18. Strength: 99.8 per cent of diethyl sulphate and about 0.1 per cent of monoethylsulphuric acid.

### APPENDIX 3

#### BENZOYLACETIC ESTER

*Principal Uses* — Intermediate for making dyestuffs.

*Equipment* — (1) Iron condensing vessel, 7 cu. m., provided with coil and iron stirrer; (2) iron separating vessel, 5 cu. m.; (3) iron stirring vessel, 2.5 cu. m., provided with coil and cooler with continuous aluminium separator; (4) clarifying press; and (5) rubber-lined precipitating vessel, 2.5 cu. m., provided with rubber-lined anchor stirrer and cooling jacket and continuous aluminium separator.

*Process* — Into the condensing vessel are introduced 1,000 l. of water, 500 l. of Naphtha C, about 130 l. of caustic soda solution (33 per cent = 180 kg.), 390 kg. of acetoacetic ester and 500 kg. of ice, the temperature being adjusted to about 5°-8°C.

Into this mixture are run in 422 kg. of benzoylchloride and 540 l. of caustic soda solution (33 per cent) with such a speed that the temperature does not surpass 15°C. before half the quantity has run in. At the end of the running in the temperature may rise to 20°C. The reaction must be distinctly alkaline to thiazol paper. The whole is stirred for another hour and then heated to

35°C. Now the whole is pressed into the vessel (2). After 1½ hr. the aqueous alkaline solution is run into the vessel (3), stirred together with 10 kg. of kieselguhr and filtered through (4) into vessel (5). The clear alkaline filtrate is mixed with 160 kg. of ammonium chloride and stirred in the closed vessel during the night at room temperature. Then about 80-100 kg. of common salt are added in order to increase the sp. gr. of the aqueous layer to 1.13. The benzoylacetate ester is allowed to deposit in the course of 2 hr. and slowly separated using the separating vessel.

The yield amounts to 415 kg.

*Recovery of the Naphtha* — The naphtha remaining in the vessel (2) is introduced into the vessel (3), distilled off with steam through a cooler, separated continuously and can be used again in the vessel (1) without drying.

The yield amounts to 475 l.

*Process Control Tests* — The sp. gr. of the aqueous layer in the vessel (5) is tested. The strength of the benzoylacetate ester is determined by ketonic cleavage by means of alkali and weighing the carbon dioxide separated from the acidified mixture.

*Raw Materials, Intermediates & Finished Product Specifications* —

Benzoyl chloride :

sp. gr.	1.211
b.p.	198°C.
m.p.	-1°C.

Acetoacetic ester : commercial product.

Naphtha C :

b.p.	130°-190°C.
sp. gr.	0.88

Benzoylacetate ester :

sp. gr.	1.118/20°C.
b.p.	149°C./12 mm.

1,000 kg. Naphtha C is obtained as by-product per ton of finished product and is used again in the plant.

### APPENDIX 3a

#### TEREPHTHALOYL bis ACETIC ESTER

*Principal Uses* — For dyestuffs.

*Equipment* — (1) Condensing vessel made of iron, iron stirrer, cooling jacket for brine, (3 cu. m.); (2) separating vessel made of iron, (5 cu. m.); (3) stirring vessel made of iron with coil and connected cooler with continuously operating separator, contents 7.5 cu. m.; (4) clarifying press; (5) rubber-lined precipitating vessel made of iron, with rubber-coated anchor stirrer and cooling jacket with connected cooler with continuously operating

separator made of aluminium (2.5 cu. m.); and (6) stone-ware suction filter with receiver.

**Process** — Into (1), containing 1,000 l. of water, 500 l. of Naphtha C and 340 l. = 348 kg. of aceto-acetic ester while stirring, 25 l. of caustic soda solution (33 per cent) are run in at a temperature of 10°C. Within 1½-2 hr., simultaneously 214 kg. of ground terephthaloyl chloride *tel. quel.* = 203 kg. of 100 per cent strength and about 355 l. of caustic soda solution (33 per cent) are introduced. The reaction must always be strongly alkaline to phenolphthalein, but not yet to Thiazol Yellow. The temperature is kept at 15°-20°C. by cooling with refrigerating brine. After stirring for further 2 hr. the mixture is separated in (2). The aqueous solution is mixed with 20 kg. of kieselguhr in (3) and filtered through (4) into (5). 68 kg. of aqueous ammonia (25 per cent) and 110 kg. of ammonium chloride are then added. Stirring is performed overnight and after further standing for 1 day the crystallized T-acetic ester is filtered off on (6).

**Yield** — 225 kg. of 100 per cent strength.

**Recovery of the Naphtha** — The naphtha remaining in (2) is distilled off in (3) by means of steam through a cooler, separated continuously and can again be introduced in (1) without being dried.

**Process Control Tests** — Terephthaloyl chloride: determination of the degree of purity by vacuum distillation.

Acetoacetic ester: commercial.

T-acetic ester: determination of the degree of purity by coupling with diazotized aniline-2:5-disulphonic acid in an aqueous pyridine solution at a pH 7 (double coupling).

**Raw Materials, Intermediates & Finished Product Specifications** —

Terephthaloyl chloride:

m.p.	75°-77°C.
b.p.	142°C./12 mm.

T-acetic ester:

m.p.	70°-71°C.
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1,900 kg. of Naphtha C is obtained as by-product per ton of finished product and is used again in the plant.

## APPENDIX 9

### BLAUSULPHOCHLORIDE

**Principal Uses** — For Blauthiosaur.

**Equipment** — Enamelled stirring vessel with jacket, contents 2 cu. m. rubber-lined, and brick-lined vessel with rubber-lined fittings and decomposing vessel, 9 cu. m. Suction filter built in masonry with wooden grate and cloth filter of 3 sq. m.

**Process** — Within 2 hr. 150 kg. of *m*-dichlorobenzene are run into the enamelled vessel containing 750 kg. of chlorosulphonic acid at 0°-10°C. Within ½ hr. the vessel is heated to 40°C., then maintained at this temperature for 3 hr. and cooled down again to 5°-10°C. Then the contents of the vessel are pumped within 3-4 hr. into the decomposing vessel which contains 1.5 cu. m. of water and 3 cu. m. of ice. After stirring for 10 hr. the sulphochloride is filtered on the suction filter and washed with ice water until neutral. The resulting product has a dry content of 80-90 per cent.

**Process Control Tests** — After finishing the sulphochlorination, a sample when placed on ice should not be oily; when adding rock salt to the filtrate, no separation of flakes should take place.

**Raw Materials, Intermediates & Finished Product Specifications** — *m*-dichlorobenzene: analysis by fractional distillation. Blausulphochloride is a slightly greyish crystalline product with a melting point of 52°-53°C. (pure: 55°-56°C.). For calculating the yield the dry content at 40°C. has to be determined.

(To be continued)

# REVIEWS

**Principles of Biological Assay**, by C. W. Emmens (Chapman & Hall Ltd., London), 1948, pp. xv+206. Price 21s. net.

THIS NOTICE OF "PRINCIPLES OF BIOLOGICAL Assay" by Dr. Emmens following the reviews I have had the honour of giving in recent issues of this *Journal* of two books on statistical methods, namely "Industrial Experimentation" (by Dr. Brownlee)<sup>1</sup> issued by the U.K. Ministry of Supply, and "Statistical Methods in Research and Production with special reference to the Chemical Industry" (edited by Dr. Owen Davis)<sup>2</sup> issued on behalf of the *Imperial Chemical Industries Ltd.*, will perhaps serve to indicate the increasing scope and speed with which publications on the use of statistical methods are now being released. For long, these methods had been used for ascertaining economic magnitudes of the resources of a country; now they are also proving their worth for improving the efficiency of agricultural and animal husbandry practices, for increasing the quantity and quality of industrial production, and for planning strategy and greater security in national defence.

In his foreword to the book, Sir Perceival Hartley, formerly of the (British) National Institute for Medical Research, where the author of this book is working, quotes with evident satisfaction a continental appreciation describing England, the home of Fisher and Yates, Finney and Irwin among several other contemporaries, as "the home of modern biometry". Rightly so. But the United States of America, too, where Chester Bliss is now working, and wherefrom come also L. J. Reed, W. R. Thompson, C. E. Rice and others, have not been lagging. After the discovery, by Professor R. A. Fisher, of the statistical techniques specially suitable for extracting *information* from *small* samples, and for testing *significance* in variations among identifiable sub-divisions (the italicized words being used in the technical sense now associated with each of them), it was only a question of time for biologists, pharmacutists and statisticians to come together in the solution of

problems arising in the production of therapeutic substances, of fungicides, insecticides and many other products in order that there may be guarantee that official requirements are complied with (up to a measurable degree at least). This has happened, sooner and more effectively perhaps than expected, because of the official efforts of the Permanent Commission on Biological Standardization of the Health Organization of the League of Nations.

The establishment by this Organization of International Standards marked a turning-point in biological assaying, and the readers of this book can follow the stages of this progress from the earliest attempts at precise measurement of potency to the varying designs for assays and in the subsequent treatment of potency, to the varying designs for assays, and in the subsequent treatment of the results. In a brief space of about 200 pages, divided into 20 practically non-overlapping chapters, suitably illustrated with worked examples, the author has admirably succeeded in writing one single book which will "enable the research worker or routine analyst who is not already an amateur statistician to plan his own tests and analyse them without reference to other manuals". The foundations are of course the methods of analysis of variance, curve fitting and regression relationship between log (dose) and response, and the first 5 chapters are occupied with these. Randomization of test objects (Latin square design) is dealt with in chapter 6, the short-cut method of using polynomial coefficients is treated in chapter 7, while chapter 8 is given to the use of co-variance analysis (rather the relative ineffectiveness of multiple co-variance analysis for increasing precision), and chapter 9 draws attention to missing terms and Bartlett's test for homogeneity. Chapters 10 to 14 deal with estimation of relative potency, standard error of M and quantal response (discontinuous variation), while "combined estimate of potency" in groups of tests and fiducial limits of error are again dealt with in chapters 17 to 20. Probit analysis is particularly dealt with in chapters 15 and 16, while the author's general appraisal to "transformations" of the response methods is probably that what he states in

1. 1948, 7, 196.

2. 1948, 7, —

para 19.5 (pages 194 and 195) — “transformations of the response may be employed to equalize variances, but are not always likely to be successful... (we) must bear in mind that it is rarely justifiable to attempt too much by way of statistical refinements... much better to repeat an assay than to spend hours trying out various transformations or tests for the rejection of aberrant responses and so forth”.

Such summary disposal of transformation methods in an otherwise admirable book is somewhat surprising seeing that much commendable work has been done using the angular transformation in biological assays. Without going too much into details (an illustration of this method appears in a paper by Knudsen and Curtis of the U.S. Federal Security Food and Drug Administration in the June 1947 issue of the *Journal of the American Statistical Association*, Vol. 42, No. 238, pp. 282-297), it may be said that the chief trouble in numerical work arises on account of the weighting factor, particularly if it involves an appeal to a normal probability curve term. In the method of angular (or square root) transformation, the weighting depends only on the number of animals used on each dose, and “a comparison of the calculation time involved shows that the probit method requires about 12 times as long as the angular transformation method involving graph and monograph” (Knudsen and Curtis). Again, in a recent paper published in September 1948 (that is after the publication of Emmens' book) in *Biometrics* (*A.S.A.*, Vol. 4, No. 3, pp. 197-210), W. R. Thompson “assays” several methods in use — the fundamental log (dose) — response curve, logistic curve, the integrated normal curve, etc., — and describes a situation in which the active agent and the inhibiting agent do not react completely but some of each remains dissociated. In his own preface, too, this author (Dr. Emmens) has stated that he confines himself only to some of the basic principles which the design of tests and the treatment of results should follow.

To be self-contained in laboratory practice, this book has reprinted Tables I to V and Table IX of Fisher and Yates' *Statistical Tables for Biological, Agricultural and Medical Research*, and, in addition, a table due to Bliss (reproduced from *Quarterly Journal of Pharm. Pharmacol.*) giving for stated values of the expected probit, the maximum and minimum corrected probits, the range, and the weighting coefficients. These tables

are, however, incorporated in the text of each chapter with numbers following table numbers of worked examples. It may have been probably more useful if they had been collected at the end of the book and marked with exclusive numbers or letters. Sometimes, as in para 4.5 (dealing with the change of base of logarithms), there is too much of (unnecessary) detail. On page 115 the limits  $0.124 \pm 0.0304 t$  is a misprint, though an obvious one, for  $0.1214 \pm 0.0304 t$ .

These minor features aside, I think this very stimulating and very effectively written book would be very welcome not only to pharmaceutical biologists, but to the orthodox statisticians also, although the author's preface opens with unnecessary modesty, with the sentence, that “this is not a text-book of statistics, or even of statistical methods of general application to biology”.

K. B. MADHAVA

**Thermodynamics**, by Edward F. Obert (McGraw-Hill Book Co. Inc., New York), 1948, pp. ix+571. Price \$ 5.50.

THIS BOOK IS INTENDED TO SERVE, IN THE words of the author, “as a fundamental text in the fields of thermodynamics and heat power. The presentation is believed to be sufficiently rigorous to satisfy the students and to serve as an introduction to more advanced topics that may be given in the later course.” The main objectives in writing the book, according to the author, are: (1) “to stress those essential aspects of the subject that every practising engineer must thoroughly understand”; (2) “to emphasize the continuity of thought leading to the Second Law”; and (3) “to show that for many real situations the perfect-gas equation provides an adequate basis for study and analysis at least for engineers who use slide rules for computations”. The text covers a wide range of subjects. It deals with numerous problems concerned with the flow of fluids, mixtures of gases and vapours, thermo-chemical calculations, air-conditioning, psychometry, and also the gas-turbine. Each chapter contains, at the end, a useful list of suggested references and also numerical problems. A list of relevant symbols is also provided at the end of each chapter.

The book undoubtedly covers a very wide field and covers it well, but it is unlikely to satisfy all the demands of a serious-minded reader interested more in “understanding the subject”, than in merely acquiring

information about it. The presentation in the text in some places gets rather heavy and involved. It has not, speaking generally, that lucidity of exposition and crispness of style which a young engineer-student would expect of a text meant to make him understand and provide insight into the fundamentals of a subject which is so important to him. Witness, for instance, the definition of *dimension* given in the opening chapter of the book. It says: "A dimension can be defined as a name describing certain qualities or characteristics of an entity. In this sense all names are dimensions and an infinite number of dimensions is possible. To reduce this number, certain descriptions may be expressed in terms of other more basic descriptions (dimensions)". An unsophisticated reader can hardly be expected to derive much satisfaction or illumination from the above definition. (One would, perhaps, have expected the treatment of dimensions to run on lines somewhat similar to that in Guggenheim's instructive article on "Units and Dimensions" in a recent issue of the *Philosophical Magazine*.) Again, though the chapter describes Reynold's number, there is no mention either in the chapter or elsewhere in the text of those important groups of dimensionless numbers that find such a wide application in the theory of natural and forced convection.

As another instance of a statement likely to confuse the student, consider the following (page 110 of the text): "The Second Law is not restricted to interchanges of heat and work, but it is rather a broad philosophy on the behaviour of energy and energy transformations. Many statements of the Second Law have been proposed; one of the most famous is that of Clausius: 'Heat cannot, of itself, pass from a colder to a hotter system'. Here, the obvious is recognized: Heat cannot flow 'uphill'; that is, the potential gradient of temperature must be in the direction of the flow of heat. The thought of Clausius can be expanded to include not only temperature, but all intensive properties (potentials): 'Energy cannot, of itself, pass from a lower to a higher potential.'" The above statement, at any rate, as it stands, is unfortunate. It often happens in science that things which, on a superficial analogy, appear to be obvious, may not really be so. In discussing the Second Law, in fact, the opportunity should have been taken to inculcate in the reader that, in spite of any feelings he may have to the contrary, the statement that heat cannot, of itself, pass from a colder to a hotter system,

is far from being obvious. If there was only one method of transferring heat, the statement of Clausius would reduce to a tautology — it would merely become a criterion for determining as to which system was the hotter and which the colder of the two. However, — and herein lies the significance of the Second Law — as there are various methods of transferring heat, the Law asserts that no matter what method one employs, heat, of itself, will never flow from a colder to a hotter body. Again, the student should have been warned in assigning any significance to the superficial analogy between temperature and potential. The concept of temperature is not analysed adequately enough in the text.

Frankly, the book is not likely to satisfy a reader who is interested in the study of the fundamentals of the subject. The book abounds in applications of the subject, provides valuable information, but for the fundamentals the reader will have to go to books by Planck, Ewing and Fermi, to mention a few names. Apart from this, the book under review will prove a useful addition to the existing large literature on thermodynamics. The charts at the end of the book, which cover a wide range of topics (for instance, the values of the total heat content of saturated air given in chart 5), will be of special interest to students of engineering and applied physics.

D. S. K.

**The Chemistry of Acetylene and Related Compounds** by Ernst David Bergmann (Interscience Publishers Inc., New York), 1948, pp. 108. Price \$ 3.00.

THE BOOK REPRESENTS A COURSE OF 3 LECTURES delivered at the Polytechnic Institute of Brooklyn in 1946. Although the author regards the book only as an introduction to a vast field, he has in fact succeeded in giving a remarkably clear and comprehensive picture of the reactions and transformations of acetylene and its derivatives. Much work has been carried out in recent years on the chemistry and technology of acetylene, which has immense possibilities as a starting material for the synthesis of a vast range of organic compounds. The applications of acetylene to the synthesis of organic intermediates, mainly due to the work of Reppe, have been described in several of the British and Combined Intelligence Sub-Committee Reports on the *I. G. Farbenindustrie*. The large-scale utilization of acetylene for

chemical synthesis has become possible by the methods developed by the I.G. for handling acetylene at high pressures and temperatures. The present book is an invaluable supplement to the technical reports, being an able and stimulating account of the mechanism by which acetylene functions in a variety of reactions. While a notable feature of the book is the very complete documentation, it is not a review, but a critical discussion which includes a considerable amount of unpublished work carried out by Dr. Weizmann, Dr. Bergmann and their collaborators.

The first lecture begins with a thermodynamical consideration of the basis for attempting to make acetylene by high temperature cracking of hydrocarbons such as methane, and proceeds to discuss the formation of diacetylene as a by-product in the German electric arc process for the manufacture of acetylene from natural gas. The behaviour of acetylene and its derivatives towards the halogens, hydrating agents, and in vinylation of alcohols, phenols and amines are then discussed. Among other reactions, an unpublished observation that acetylenic alcohols add to diene hydrocarbons to give alcohols of the dihydrobenzene series is mentioned. The metal derivatives of acetylene are finally reviewed.

The addition of nitrogen compounds to acetylene is further elaborated in the second lecture. Charts of Reppe's acetylene-formaldehyde and acetylene-acetaldehyde condensations are accompanied by a treatment of the reactions and products. Weizmann's studies of the reaction of acetylene with ketones, using as condensing agent potassium hydroxide dispersed in an acetal or a dialkyl ether of an ethylene glycol, and of the transformations of the acetylenic carbinols and diols are described. The lecture concludes with a reference to the fact that acetylene provides us with all the substances, except for isobutylene, required for producing the known synthetic rubbers.

The third lecture commences with a further discussion of the properties of the system potassium hydroxide plus solvents such as acetal or glycol dialkyl ethers. The polymerization of acetylene is then considered, and the mechanism of the formation of vinylacetylene, divinylacetylene and the higher polymers is explained. From the reactions of various acetylenic compounds it is shown that the triple bond can participate in a conjugated system with a double bond to cause resonance phenomena. The polymerization

of acetylene to cyclo-octatetraene and the properties and reactions of the latter are discussed. There is a brief account at the end of acetylene derivatives occurring in nature. K. V.

**Paramagnetic Relaxation**, by C. J. Gorter (Elsevier Publishing Co. Inc.; Cleaver-Hume Press Ltd., London), 1947, pp. vii+127. Price 12s. net.

THE BEHAVIOUR OF PARAMAGNETICS IN STATIC magnetic fields has been the subject of extensive study since the days of Faraday. Both theoretical and experimental work has laid, well and truly, the foundations of our knowledge of this branch of magnetism.

The study of the properties of paramagnetic substances in a magnetic field which varies harmonically (with frequencies of the order of  $10^6$  Hz.) has been comparatively of recent origin. Investigations lead to 2 terms. The first is the high frequency magnetic susceptibility whose dependence on the applied frequency is called paramagnetic dispersion. The second term characterizes the paramagnetic absorption of energy from the high frequency magnetic field. Waller's paper in 1932 first gave a detailed theory of paramagnetic relaxation. He discussed in this paper the dependence of relaxation time on the strength of the magnetic field and temperature.

Extensive experiments have been carried out on paramagnetic absorption and dispersion by 3 independent methods. The first is the heterodyne beat method developed mostly in Leyden; the second method is a calorimetric one used extensively at Amsterdam. Finally, detailed investigations have been carried out at Leyden by the bridge method for the simultaneous determination of paramagnetic dispersion and absorption.

In the book under review, these problems have been treated in detail. The first chapter gives a historical account of the subject and makes clear the fundamentals involved in the study of paramagnetic relaxation. Thermodynamical considerations lead to a relation between adiabatic magnetic susceptibility and ordinary or static susceptibility. At low frequencies the susceptibility tends to the static value while at high frequencies the adiabatic value is obtained.

The second chapter gives full details of the 3 experimental methods stated above. The advantages and disadvantages of each of these methods are fully discussed. A review

of the results obtained by these methods with several substances is given in chapter III.

A résumé of the theory in the light of experimental evidence is presented in chapter IV. The theories of spin and lattice relaxations are considered in detail. The exchange of energy between the magnetic ions and the lattice is considered either as a lattice vibration quantum absorbed or emitted by the ions or alternatively as a vibration quantum being scattered non-elastically by the magnetic ions — a sort of a "Raman" process. The first order process preponderates at liquid helium temperatures while "Raman" processes are more frequent at liquid air temperatures. These theoretical considerations constitute the basis of the theory of paramagnetic relaxation.

Chapter V presents a discussion of the results outlined in chapter III on the basis of the theory given in chapter IV. It is well recognized that a qualitative understanding of the phenomena involved has been reached in certain respects. Several problems which need further study and elucidation have been briefly stated at the end of the book.

The book under review presents, in a concise and lucid form, full theoretical and experimental details of this new and rapidly growing branch of paramagnetism. Investigators in the field of magnetism and allied subjects are sure to find much in the book that would be of special interest and importance.

S. R. R.

**Colorimetric Methods of Analysis**, Vol. I, 3rd edition, by F. D. Snell & C. T. Snell (D. Van Nostrand Co. Inc., New York; Macmillan & Co. Ltd., London), 1948, pp. xii+229. Price \$ 4.50 or 25s. net.

THE BOOK UNDER REVIEW IS THE THIRD edition of the first volume of the authors' comprehensive treatise on *Colorimetric Methods of Analysis* and includes some important nephelometric, turbidimetric and fluorometric methods. The latest edition of the entire treatise is divided into three volumes. The present volume, the first of the series, covers treatment of theory, instruments and  $pH$ . The second and third volumes deal respectively with inorganic and organic applications of the methods.

The treatment of the subject-matter in this volume is rather of a condensed nature, as it aims at a complete treatment of all instruments and methods. As such, it will

not be easy for those, who are not already more or less familiar with these methods and instruments, to follow their description for practical work. But as more or less complete and up-to-date references to original literature have been cited in all cases, those interested in the subject will undeniably find the book to be of great help.

Colorimetric methods, together with its newer development in the form of nephelometry, turbidimetry and fluorometry, are acquiring ever-increasing usefulness and importance as powerful and speedy means for the estimation of minute quantities of substances not only in the field of chemistry, but also in various other allied branches of science, particularly in biology, physiology, soil science, etc. In the assay of vitamins and certain other biological materials, colorimetric methods are the only available means of investigation. They are also being extensively used as controls in the manufacture and standardization of many industrial products related particularly to foods and drugs. The present volume will, therefore, not fail to receive a wider welcome than what might have otherwise been restricted to the circle of analytical chemists only.

After a general but brief account of the various types of colorimetric methods, a short discussion of the essential theory behind these methods, dealing with the physical laws of transmission and absorption of light, has been added. This is followed by a somewhat detailed account of the various methods including spectrophotometry, filter photometry, and photo-electric methods together with a short description of the various types of instruments employed in each case. An entire chapter has been devoted to the description and use of glass filters. Then an account of nephelometry, turbidimetry and fluorometry with a description of the different instruments employed for the purpose has been added. A chapter hereafter deals with the factors limiting the accuracy of colorimetric methods and the typical sources of errors involved. Then follows a short chapter on calculations for different types of methods. A considerable part of the book has, however, been reserved at the end for the discussion of colorimetric methods for determining hydrogen ion concentration or  $pH$  values of solutions with the help of indicators. This includes an account of the theory underlying the methods and deals with the sources of errors and accuracy in each case; preparation of various buffer

solutions and indicators have also been included, followed by a somewhat detailed description of the preparation of samples for pH determination; the book then closes with an account of the different methods of pH measurement describing the procedure to be followed under each.

Author and subject indices at the end have been carefully prepared. The printing and get-up leave nothing to be desired.

There are a few misprints or errors which have come to the notice of the reviewer. Of these, the following may particularly be mentioned:

On page 65, "transmittance= $I_1/I_2$ " should be inverted and written as  $I_2/I_1$ .

On page 143, 1.14, "0.1000" should be 1.0.

On page 159 in eq. (11) at the bottom, " $(1-\alpha)K_m$ " should be replaced by  $\alpha K_i$ .

On page 210 in the last line over Table 44, the words "sample" and "standard" should better be exchanged; the same holds good also for the words "initial" and "final" on page 217, in line 8 from the bottom.

The deduction of eq. (13) on page 161 is also not quite clear.

P. RAY

**Handbook of Textile Industry**, by Sir Padampat Singhanian (Bureau of Economic Research, Kanpur), 1948, pp. xxxii + 542 + iii. Price Rs. 15.

INDIA'S PREMIER INDUSTRY — THE TEXTILE industry, is by far the single largest industry of the country with a block capital of Rs. 100 crores. The industry employs nearly a million people in its different stages. The total value of the annual production by the industry exceeds Rs. 400 crores. All the above facts are exhaustively annotated in this handy publication.

This collective publication compiled by a group of economists, technicians and industrialists and edited by the Director, *Bureau of Economic Research*, Kanpur, is comprehensive and up to date from every point of view — statistical, financial and technical.

In addition to cotton textiles, the book contains an authoritative account of other textile materials: woollen, silk, jute, flax, rayon, etc. Auxiliary requirements for the industry such as mill stores, machinery, spare parts, chemicals are dealt with in the publication.

The chapter on "Post-war Planning" is a lengthy one, and includes summaries of Industrial Panel Reports, both published and unpublished. The statistical section is an elaborate one and gives several useful tables.

The financial aspects of the industry have received special attention. The balance-sheets and working results of over 500 leading textile concerns (for periods extending from 4 to 7 years) are critically analysed. Other topics discussed under this section are cotton consumption, labour employed, capital structure, profit earning, etc. This chapter will be of special interest to the investing public.

The "Who is Who" section with specially prepared write-ups and photographs gives biographical sketches of all those personalities who have contributed to the growth and stability of the textile industry. The proceedings of the last four sessions of the *All-India Textile Conference* with special emphasis on some of the important pronouncements made during the conference by leading textile technicians and industrialists are included.

The Handbook is an indispensable reference volume to all connected with the textile industry.

A. K.



# NOTES & NEWS

## Estimation of Lactose

THE EXISTENCE OF UNCERTAINTIES in the tables employed in the determination of lactose, alone and in the presence of sucrose, by the method of Munson and Walker has led to the redetermination of the sugar-reducing values from which a set of tables have been computed (*J. Res.*, 1948, **41**, 211). The copper equivalents of lactose determined range from 7.7 mg. up to 342.0 mg. alone and in the presence of 6.6 mg. to 355.8 mg. of sucrose. The method employed is that of Munson and Walker (*J. Amer. Chem. Soc.*, 1906, **28**, 663).

The lactose used was prepared from U.S.P. lactose. A hot 50 per cent solution was treated with vegetable char and filtered. Aluminium hydroxide was then added, solution filtered and lactose crystallized. The material was re-crystallized, washed with water at 5°C. and dried at room temperature in a desiccator. Analysis showed: moisture, 5.05 per cent; ash, 0.003 per cent; and  $[\alpha]_D^{20} = 52.54^\circ$ . The data indicate that the material is lactose monohydrate,  $C_{12}H_{22}O_{11} \cdot H_2O$ . The sucrose used was National Bureau of Standards, sample 17.

The basic conditions for the determination, as given by Munson and Walker, were followed. The Soxhlet reagent contained 34.639 gm. of copper sulphate,  $CuSO_4 \cdot 5H_2O$ , in 500 c.c., and 173 gm. of potassium sodium tartarate ( $KNaC_4H_4O_6 \cdot 4H_2O$ ) and 50 gm. of sodium hydroxide in 500 c.c. All solutions were measured at 20°C.

In the original method the solution was heated over a gas flame, but in this work electrical heating was substituted. The heater was such that the solution in the beaker was practically surrounded by the heating element and the current was controlled by a constant voltage regulator and a variable ratio transformer. The solution could be brought to boil in the required time within  $\pm 5$  sec.

The determinations were made as follows: 50 c.c. of the solution containing lactose were transferred to a 400 c.c. beaker containing 50 c.c. of the mixed Soxhlet

reagent. The solution was heated to the boiling point in 4 min. and then allowed to boil 2 min. longer. The cuprous oxide was transferred immediately to a Gooch crucible and washed with water at 60°C. It was then dissolved by adding 5 c.c. of 1:1 nitric acid, and the crucible was quickly covered. Before the addition of acid a small quantity of water was added to the crucible to minimize the evolution of nitric oxides. The copper nitrate and washings were received in a 250 c.c. beaker to which 10 c.c. of 1:1 nitric acid and about 5 gm. of ammonium sulphate had been added. Sufficient water was added to cover the cylindrical platinum-gauze electrodes, the total volume of electrolyte being about 180 c.c. The electrolysis was conducted overnight at room temperature and at an approximate current density of 0.10 amp./dm<sup>2</sup>. Upon completion of the deposition, the electrolyte was replaced by distilled water before the circuit was broken. The copper was washed with alcohol, dried for 15 min. at 100°C., cooled in a desiccator and weighed. All deposits were bright and showed no evidence of oxidation.

## Controls for Testing Textile Colour Fastness

A SIMPLE METHOD OF CONTROLLING the integrated exposure of fluctuating arc lamps employed in testing light fastness of textile dyeings is described (*J. Res.*, 1948, **41**, 109). The procedure is to expose a piece of light-sensitive test paper, placed in front of an arc lamp along with materials to be tested, and terminating the exposure when a match with a standard, observable with the unaided eye, is obtained. The standard is a strip of the same batch of paper that has been exposed in a master lamp to a definite light dosage.

Carbon arcs are commonly employed for testing colour fastness of dyed fabrics and of stability of materials in general to light. The lamps usually employed are "Fade-O-meter" type. Since uniform performance of arc lamps is a practical impossibility, a method of exposure control by

estimating the radiant output with a light-sensitive material is a necessity. The method, based on standards produced with reproducible light dosages rather than reproducible batches of the light-sensitive media, has been found satisfactory. Freedom from difficulties of batch reproducibility, ease of recognizing the extent of exposure, reasonable assurance of uniformity within each batch, wide range of exposure periods available, and general standardization of procedure, constitute the main advantages of the method.

A special kind of paper was coated with a light-sensitive dye. A portion was standardized in a master lamp by exposing it to a convenient measured light dosage defined in terms of "standard arc hours". This standardized paper becomes a secondary standard to gauge light dosage in other lamps. In a typical application, a piece of light-sensitive paper is simultaneously exposed with materials to be tested, and when a match is observed with the unaided eye, the materials are assumed to have received corresponding light dosages. Estimating the degree of exposure is facilitated by auxiliary standard strips representing 80, 90, 110 and 120 per cent of the light dosage.

The actual time of exposure is immaterial unless it differs consistently by 30 per cent or more from the number of standard arc hours identified with the exposure, indicating that an adjustment of the lamp may be desirable.

The requisites of a light-sensitive paper are: colorimetrically stable in the dark, both exposed and unexposed samples producible in large uniform batches, normally responsive to temperature, arc characteristics and relative humidity, and visually sensitive.

2 samples produced at the National Bureau of Standards were found suitable. The papers were neutral (pH 6.8) and unsized. Paper 1444 was made entirely of wood pulp and paper 1370 entirely of cotton. The papers made from cotton fibres were considerably more uniform in fading rate than those made from wood pulp. These 2 papers were tested with 47 representative dyes and it was found that National Aniline Niagara Blue G. Conc., lot 64704, colour index 502, and, to a lesser extent, Du Pont Victoria Blue Conc., lot 22, colour index 729, gave the most useful products. By varying the dye, fibre and method of

application, papers of different fading rates were obtained. The samples had the following treatment and exposure characteristics:

(a) Paper C: Exposure time, 3 to 10 hr.; paper 1370, tub-dyed with 0.09 per cent Victoria Blue in 1:3, 95 per cent ethanol tap water solution.

(b) Paper E: Exposure time, 10 to 40 hr. Paper 1370, tub-dyed with 0.54 per cent Niagara Blue in tap water.

(c) Paper F (or A): Exposure time, 40 to 100 hr. Paper 1444, tub-dyed with 0.65 per cent Niagara Blue in tap water. A somewhat lighter modification of paper F has been referred to as paper A.

(d) Paper S: Exposure time, 20 to 40 hr. This paper was beater-dyed by adding a solution of Niagara Blue to give a 0.5 per cent ratio dye: fibre, using the same kind of fibre as in paper 1444. Melamine resin (3 per cent on fibre weight) was added to the beater to give high wet strength for use in lamps with water spray.

These 4 types serve to indicate the methods of producing papers of various desired characteristics. The tub-dyed papers, which are finished papers run through a dye bath, must be used dry; the beater-dyed paper is non-leaching and may be used for "weathering" lamps, but is decidedly inferior to the tub-dyed papers in sensitivity. The latter have the disadvantage of tending to exhaust the dye bath which thus requires replenishing from time to time.

All the samples were uncalendered after dyeing and were found unchanged during storage for 8 to 18 months.

The standardized strips are mounted in a comparison booklet having flexible black cellulose acetate covers to protect the strips from soiling and light during non-use.

Although temperature has no effect on the fading rate of papers in the dark, it has considerable influence during exposure to light. Temperature specifications for papers should ensure uniformity of their performance. The light-sensitive papers showed excessive fading whenever exposure was interrupted through failure of arc in overnight runs. Other factors requiring standardization are: relative humidity, type of arc, and voltage.

With the master lamp it is not possible to obtain a precise fraction of a light dosage for auxiliary

standard strips because of variation of electrode resistance during the burning of carbon electrodes. For this reason light dosage is measured by means of an "exposure meter".

For reproducible results the carbon electrodes must be of uniform quality and size. The arc is enclosed in a 18 cm. Corex-10 tube, and the arc chosen is one which burns in a current of air without depositing any residue. The arc lamp is housed in a special room at  $25 \pm 0.5^\circ\text{C}$ . and  $50 \pm 1$  per cent relative humidity in order to avoid seasonal effects. Regulation of arc current is necessary for reproducible performance of the arc. A device, which is essentially a contact-making ammeter, regulates the arc current at 30 amp. by controlling a reversible motor that regulates the arc gap. The line voltage is kept within a tolerance of  $\pm 0.5$  per cent by means of an induction voltage regulator, mechanical type. The line voltage is applied to a fixed transformer that operates the arc near 40 volts.

By limiting the tests to a definite portion of the electrodes, it is possible to obtain a measure of the reproducibility of the arc by relating radiant energy which was measured in turn by an exposure meter.

## Synthetic Methanol

RECENT DEVELOPMENTS IN SYNTHETIC methanol production in the United States have been reviewed (*Ind. Eng. Chem.*, 1948, 40, 2230). The phenomenal growth of the industry is attributed to the accelerated demand for formaldehyde required in the plastics industry. 44 per cent of the output (total output 138 million gal. in 1948) is utilized in formaldehyde production and 38 per cent in the automobile anti-freeze industry.

The more important synthetic methanol plants are the *Du Pont* plant located at Belle, W. Va., and the 2 plants of the *Commercial Solvents Corporation* at Peoria and at Sterlington. The *Du Pont* unit, a by-product unit attached to the ammonia synthesis plant, was the first to go into operation. The carbon monoxide impurity was removed by methanation. This practice of running a single synthesis gas and steam through both methanol and ammonia converters is considered to be a major American contribution to the technology of methanol syn-

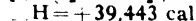
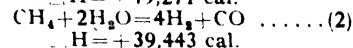
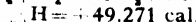
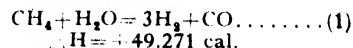
thesis. This practice is widely adopted in Europe.

The *Commercial Solvents Corp.*'s plant, also a by-product unit, uses fermentation gas from butyl alcohol-acetone units as raw material. The gas consists of carbon dioxide (60 per cent) and hydrogen (40 per cent). This installation was the first to use high pressure synthesis employing a specially developed catalyst. The Sterlington plant also operates on the same lines.

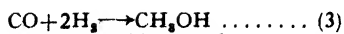
The raw gas for the Sterlington plant is obtained from the nearby Monroe gas-fields (about 50 miles) at a pipe-line pressure of 35-50 lb./sq. in. The gas is made up of methane (93.18 per cent) and unsaturated hydrocarbon gases. The gas is fed to the reformer furnace at a pressure of 27 lb./sq. in. The process gas is pre-heated in a gas-fired spiral tube ( $225' \times 5"$  dia.) and passed through sulphur removing drums ( $6' 6"$  dia.  $\times 28'$  deep) filled with extruded zinc pellets ( $0.1875' \times 0.5"$ ). The purified gas is mixed with steam at 30 lb./sq. in., which has been heated to  $940^\circ\text{F}$ . in a shell and tube heat exchanger. The ratio of steam to gas is mechanically adjusted, and mixing is accomplished by a simple T-joint. The combined gas and steam passes downward through a bank of 132 catalyst-filled tubes arranged in 6 sections in the reformer furnace. The catalyst consists of 0.75" ceramic cubes impregnated with finely divided metallic nickel. The furnace is gas-fired from the top and held at about  $1,750^\circ\text{F}$ . The flow of gas to the furnace is regulated by the temperature reading of a thermo-couple located in the reformed gas outlet where the temperature is held at  $1,250^\circ\text{F}$ .

The reformed gases pass immediately through a steam superheater where the temperature drops to about  $950^\circ\text{F}$ , then into a boiler feed water-heater where it is cooled to  $335^\circ\text{F}$ . and finally in a bubble-cap tower cooler. The water stream in the cooler is controlled to hold the temperature below  $120^\circ\text{F}$ . to avoid corrosion.

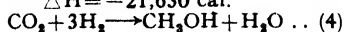
The reaction of steam and methane in the reformer follows the equations:



From these reformed gases methanol is formed according to the following equations:



$$\Delta H = -21,630 \text{ cal.}$$



$$\Delta H = -11,850 \text{ cal.}$$

For the products of equation (1) and (2) to react according to equations (3) and (4), excess carbon oxides have to be provided. At the Sterlington plant excess carbon dioxide is introduced along with the reformer gas. This procedure is wasteful as a third of the hydrogen generated is converted to water and methanol gets diluted. The recent practice is to exclude carbon dioxide and induce the reaction to proceed according to equation (3), the only drawback being excessive temperature in the catalyst mass which, if not properly regulated, results in methane reformation. Addition of carbon dioxide in the converter feed has a quenching action and affords a simple means of controlling the speed and temperature of the methanol synthesis reaction. Synthesis from monoxide gives about 95 per cent methanol, and the mixture of dioxide and monoxides gives about 80 per cent methanol.

The methanol converters contain catalyst-filled baskets of a special design patented by the *Commercial Solvents Corp.* They are so designed that the baskets can be lifted from the top of the converter shell by an overhead crane for convenient examination and replacement. The catalyst is a specially patented product by the *Commercial Solvents Corp.* and was developed first at the Peoria Works (French Patents: 635,023 and 636,337, 1927). Since the converters are estimated to have about 12 to 15 per cent conversion per pass, new make-up gas comprises only about 15 per cent of the feed.

The Sterlington plant, operating at about 5,000 lb./sq. in. and 400°C., is a relatively high temperature and low pressure operated unit. Current practice in methanol conversion makes use of 7,200 to 14,700 lb./sq. in. pressure and 250° to 400°C. temperature. The percentage conversion varies inversely with the temperature and directly with pressure. Optimum conditions are reported in the temperature range of 300° to 400°C.

Condensed methanol and unreacted gases from the after-cooler flow to a methanol separator where separation of the gases and methanol is accomplished and liquid methanol flows by mechanical liquid level control to the let-down drums from where it is

pumped through positive displacement meters to intermediate storage tanks.

An external refrigeration system, having a capacity of 130,000 B.T.U. per hr., is provided to condense the vapours vented from the let-down drums.

The crude methanol is distilled in a continuous refining unit consisting of 3 columns, the 1st one being a 45-tray column. Crude methanol, pre-heated, is introduced between the 17th and 24th tray in the 1st column. The column is heated by 35 lb. steam through a closed calandria cut in below the last tray. The 3rd column is the largest consisting of 75 trays; the feed is introduced at the 14th and 19th tray, and methanol is taken off between 67th and 73rd tray.

Extensive instrumentation and automatic controls are a feature of the plant. Most gas flow meters operate by measuring the pressure drop through a fixed orifice. Automatic valves are of the air-actuated diaphragm type. Temperature measurement is by thermo-couples. Pressures are measured by Bourdon tubes. All compressors are equipped with automatic cut-outs operated by lubricating oil pressure as well as emergency switches located at strategic points in the compressor house. Any or all of the compressors can be stopped instantaneously. A safety device also is installed on the reforming furnace which actuates an alarm if the furnace temperature exceeds 1,500°C. Automatic liquid level indicators are of the mechanical float type. Bubbler type direct reading indicators are used to indicate still reservoir levels.

### Industrial Chromatography

CHROMATOGRAPHY HAS NOW become a commercially feasible unit operation (*Chem. Met. Eng.*, 1948, 55, 133). In the separation of rare earth elements by this method, it is possible to separate the individual elements with high purity so that they contain less than one part per million of either of the adjacent rare earth neighbours.

The possible uses for chromatography are: (1) testing the homogeneity of a substance; (2) establishing the identity or non-identity of 2 substances; (3) concentration of a product occurring in natural sources at great dilutions; (4) separation, identification, estimation and isolation of the constituents of a

mixture; (5) purification, e.g. of a technical product; and (6) identification and control of commercial products.

The commercial adsorbents commonly employed are: activated carbon, alumina, silica gel, sugars, aluminium oxy-hydrate, magnesium oxide, calcium hydroxide, calcium sulphate, calcium carbonate, soda ash, Floridin, Fuller's earth, and the zeolites. The ion-exchange resins exhibit the same chromatographic separability that the physical adsorbents do. Moreover, these can be tailor-made for an individual job.

The design of chromatographic columns for batch operations is determined by the flow rate, particle size, column diameter and column height. The main calculable factor is pressure drop. The adsorption columns used by the *Valley Vitamins Inc.*, Tex., are 26" in dia. and 7' in height. Adsorbent supports consist of: first, a steel plate with  $\frac{1}{4}$ " holes drilled on  $\frac{1}{4}$ " centres, then a 100-mesh stainless or monel screen, and finally a  $\frac{1}{4}$ " galvanized screen. Columns used by *Merck & Co.*, at Elkton, Va., are 36" in dia. by 12' of packed height. Bed supports for these columns consist of sieve plates and layers of fine mesh wire cloth. Pressure drop through these columns is 60 psig.

$\beta$ -carotene, xanthophyll and chlorophyll are produced from alfalfa by *Valley Vitamins Inc.* The ground alfalfa is first extracted with hexane and the 3 pigments separated by chromatography. The resulting separate solutions are concentrated, and the products recovered for sale. 6 adsorption towers are in service. Normal practice is to charge the towers in pairs, each pair being charged in parallel. At a given time, one pair is being charged with the crude solvent mixture, the second pair is eluting xanthophyll and the third pair is eluting chlorophyll. Carbon black is the adsorbent employed. Hexane is charged at the bottom at a rate of 3 gal. per min. at first, and then the crude extract at the rate of 1 gal. per min. Operation is continued till lipoids appear in the discharge. The time taken is 126-155 min. Sight glasses are provided at the top of the tower. The charging of extract is stopped when the yellow colour of the carotene appears in the discharge. Hexane is pumped next for about 110 min. till the yellow colour of the discharge begins to fade.

The towers are next allowed to drain to avoid contamination by hexane of the hexane-isopropanol solvent used for the elution of xanthophyll. The solvent is pumped from the bottom at the rate of 2 gal. per min. The darkening of the sight glasses shows that xanthophyll has started eluting. The time taken is about 4 hr. for the xanthophyll to elute, which is indicated by the fading of yellow colour.

Benzene-isopropanol mixture is employed for eluting chlorophyll which is pumped from the top. The discharge is for about 8 hr., the bottom sight glasses showing a light green discharge. The tower is washed for another 8 hr. with fresh benzene-propanol thus completing the cycle.

Carotene extract which is contaminated with lipoids is subjected to a second adsorption step for the removal of lipoids. 4 more towers are employed for this purpose. Fresh hexane is pumped into the bottom of 2 towers at about 3 gal. per min. and when the solvent is flowing out through the sight glasses, the carotene extract is pumped in at the same rate until the lipoids appear in the discharge (tested manually by the greasy character of the discharge fluid). The feed rate is then reduced to 2 gal. per min. until a yellow colour appears in the discharge which takes about 45 min. Weak carotene eluate is pumped through until the yellow colour fades. The towers are washed with benzene-isopropanol solvent till the discharge shows no yellow colour and finally with hexane for about 90 min. to complete the cycle.

Merck & Co. uses chromatographic adsorption on alumina to produce highly active streptomycin. When a solution of crude streptomycin is passed through the column and then eluted with pure methanol, the activity of the effluent rises rapidly from 0 to a peak, and then tails off slowly. A solution of streptomycin hydrochloride in methanol is pumped through the columns at 60 psig. pressure into the top of the column, which is fitted with 100-200 mesh alumina. At the end of a given amount of feed solution, the pumping is stopped and the column is fed with pure methanol to elute the crude streptomycin at the top of the column. The effluent from the column is tested chemically by spot tests and when active material appears, the effluent is directed to

a receiver directly under the column. Spot chemical analysis is permissible despite the time it takes, because the entire cycle takes 24 hr. The column is re-vivified with distilled water. Channelling and faulty column operation is avoided by maintaining the column full of liquid at all times.

At present 2 major fields of the process industry — biochemical and petroleum refining — are actively employing chromatography. The biggest advance in the petroleum field would be the development of continuous chromatography. The up-grading of lubricating oil fractions by removal of aromatic constituents, and the production of pure organic chemicals from natural, cracked hydrocarbons, are 2 fields in which chromatography is likely to be employed on a large scale in the future.

#### Improved Methods of Sulphuric Acid Manufacture

A NEW THEORY OF SULPHUR dioxide oxidation has been recently developed and embodied in several patents by the Italian chemists: Guareschi, G. Maragliano-Buseti and Lucia Pettinati (*Chem. Age*, 1948, 59, 849).

An improved method has been envisaged for the production of oleum by catalysis, whereby plant and operation are simplified and considerable economy in cost and space is ensured. In this invention, the mixture of gases from the pyrites furnace, after purification, is liquefied. The mixture is reduced to  $\text{SO}_2 + \frac{1}{2}\text{O}_2$  ratio and led into a heat recuperator attached to the pyrites furnace where the temperature is raised to 450°-480°C. Owing to the high concentration of gas, catalytic reaction takes place giving a high yield.

In another patent on oleum manufacture, instead of the  $\text{SO}_3$  being absorbed by the acid in a gaseous state, it is solidified and dissolved in this form in a mono-hydrated sulphuric acid (98 per cent) solution. By this all bulky and costly plant parts, such as absorption towers, are eliminated. In the patent by Maragliano-Buseti and Pettinati for the intensive manufacture of sulphuric acid, nitrogen oxides are used as reaction catalysts and accelerators. Sulphuric acid is formed simultaneously with the recovery of liberated nitrogen oxides. The  $\text{SO}_3$  is in intimate contact with nitro-sulphuric acid (nitrosyl sul-

phate) dissolved in sulphuric acid of over 60°Be. The reaction is at first vigorous and nitrogen oxides are set free in bulk and are oxidized immediately. By suitably varying the amount of nitrosyl sulphate, the composition of sulphureous gas mixture and the temperature, the two phases involved in the cycle become practically simultaneous.

#### Germanium Electronic Appliances

A GERMANIUM CRYSTAL RECTIFIER, which has been recently developed, is less liable to overload than the usual silicon rectifier (*Nature*, 1948, 162, 982). It can withstand a peak inverse voltage of 80 volts and deliver a maximum continuous current of 50 milliamperes; although its frequency response is inferior to that of silicon, it is very much superior to that of the metal oxide rectifier. As a detector or rectifier, it can replace the vacuum diode with considerable advantage being a non-heating device. A.C. hum, noise and thermal delay, from which all hot-cathode devices of the vacuum type suffer, are absent. The germanium crystals used in these high back-voltage crystal rectifiers are obtained from ingots formed *in vacuo* and slowly cooled.

A new 3-element germanium electronic device, termed "transistor" (transfer resistor) or semiconductor triode, has been developed at the *Bell Telephone Laboratories*. The device consists of a metal cylinder about an inch long and of the thickness of a pencil containing germanium and its 3 electrodes. The transistor is used to amplify telephone and television signals, and a standard model radio receiver with transistors in place of the ordinary valves, operates quite satisfactorily. A standard frequency note generated by an oscillatory circuit employing transistors has also been produced. The transistor uses less power than a vacuum tube, has an output of 25 milliwatts and can operate at frequencies up to 10 megacycles per sec. Owing to their simplicity, small size, performance, long life and probably low cost when mass-produced, these transistors should find many applications in all forms of electronic equipment.

The preparation of germanium of rather unique properties, of which the most striking is the absence of surface rectification at the germanium-metal contacts,

has been recently announced from the G.E.C. Research Laboratories. The germanium ingots were prepared by melting germanium powder under a pressure of less than  $10^{-4}$  mm. of mercury. Its high magneto-resistance and variation of Hall Coefficient with magnetic field, in spite of the high degree of homogeneity, is a point of great interest and value in the designing of electronic equipment.

### **Spectral-transmissive Properties & Use of Eye-protective Glasses**

CONSERVATION OF EYESIGHT is one of the foremost problems confronting safety engineers today. They have found that protection of the eyes from excessive exposure to radiant energy is as important as protection from mechanical injury. Information on the transmissive properties of most of the widely distributed makes of tinted lenses is now available in a new circular, *Spectral-Transmissive Properties & Use of Eye-Protective Glasses*, recently issued by the National Bureau of Standards, U.S.A.

This publication is the result of a study of the spectral-transmissive properties of about 200 glasses intended for use either as sun-glasses or in special industrial operations where injurious amounts of radiant energy are present. Extensive consideration is given to elimination of glare, colour distortion, use of glasses for night driving, standardization of glasses for outdoor and industrial purposes, and the spectral-transmissive properties of glasses for use under various types and intensities of ultra-violet, luminous, and infra-red radiation. It is proposed that sun-glasses be grouped into three or four shades having luminous transmittances of 60, 35, 25, and about 10 per cent respectively, values that correspond roughly to those for the industrial shades numbered 1.5, 2.0, 2.5 and 3.0.

### **A Measuring Device for Radioactivity**

THE DEVELOPMENT OF A NEW and compact device for measuring radioactivity was described by Dr. Benjamin Schloss of the Nuclonic Sales Co. at a symposium of the American Chemical Society at New York. The new instrument excels the Geiger counter and is expected to facili-

tate the use of tracer isotopes in medical research (*Chem. Age*, 1948, 59, 820). Described as a "crystal counter", the instrument utilizes a small crystal to trap and detect radiation in place of the comparatively bulky gas chamber of the Geiger counter. It efficiently records  $\gamma$ -rays.

### **Process Development Laboratories**

THE 4 REGIONAL RESEARCH Laboratories operated by the U.S. Department of Agriculture, specially designed for process research, are in operation since 1940-41. The experiences of the chemical engineering staff in these institutions and their opinions regarding the design and operation of process development areas are described in a recent number of *Industrial & Engineering Chemistry* (1948, 40, 1014).

These laboratories were established with the specific purpose of applying the results of scientific and technological research to the utilization of farm commodities. Research is not restricted to the discovery of new uses for farm products, and considerable research on the improvement of established means of utilization is also carried on. A sum of \$4,000,000 is expended on these laboratories yearly. Each laboratory maintains a total staff of 300 persons, approximately 200 of whom are scientists and engineers of professional grade. The buildings are designed to house a research staff of this size.

As large-scale commercial application of research results is regarded as the criterion of success, emphasis has been laid, during the planning of buildings, on facilities for process development on a pilot-plant scale. From the first it was evident that no one plan for the pilot-plant space would serve equally well for all the 4 laboratories.

The laboratories are all 4-storey buildings constructed on the U-plan. The base of the U contains the administrative offices. One of the long wings consists of typical research laboratories, and the other the pilot-plant area. The length of the wings vary from 256' to 304' but the width is maintained constant at 64'. Because of special differences in research programmes, no 2 industrial wings are developed alike. All the laboratories, however, contain a large open area, 4 storeys in height, measuring 45' from floor to roof beams.

The pilot-plant area proper measures 112' to 208' in the different laboratories.

In the Northern Laboratory, space is set apart and partitioned for housing an alcohol pilot plant and this has a floor area of 96'  $\times$  64'. In addition, a separate specialized pilot-plant building has recently been erected to house large-scale experimentation units on production of motor fuels and other products by hydrolysis of agricultural residues such as corn cobs.

In the Southern Laboratory, experimental textile equipment is installed on 3 one-storey floors, each 96'  $\times$  64', and cotton treating equipment including kier boiler, calender, dryers, etc., are housed in the chemical wing (96'  $\times$  26').

After 7 years of varied operational experience, the engineering staff of these laboratories are of the opinion that 45' head room in the pilot-plant room is excessive and 22' are ample for the majority of operations. Another lesson is that distinctly different types of large-scale development cannot be successfully carried on together in the same room. In the Western Laboratory, large-scale food processing and chemical process developments were for some years located in the general pilot-plant area. This has been far from satisfactory. The design requirements for a chemical process room and food processing room are different. The latter must be built so that scrupulous cleanliness is not only possible but relatively easy to attain. Foreign odours, dust and fumes must be excluded.

The general pilot-plant areas are constructed at ground level with heavily reinforced concrete floors and direct truck access through a roll-up door for which a width of 15' is recommended. The solvent extraction plant is treated as an explosion hazard. In all the laboratories the 35' span between rows of columns is bridged by a 5-ton electrically operated travelling crane which has proved very useful for moving heavy equipment.

Provision for the supply of utility services to these areas was given most careful thought. High pressure steam (120 lb.) in a 2.5" line, city water in a 4" line, gas in 4" line are carried most of the way round the area. Shut-off valves in the loops permit isolation of a section during installation of a new connection without shutting down the entire area. Plugged tees at 16' intervals along the

loops make it easy for new connections.

The primary electric supply to the area is a heavy 208-v., 3-phase bus of 2,500 amp. capacity carried all the way around the walls in a grounded metal box below the second floor level. Heavy connections are made direct from the bus. Load centre boxes fed from the bus are mounted at intervals on the walls and conduit lines are run from the nearest load centre to lighter equipment. In addition, heavy duty single-phase and 3-phase plug receptacles are installed on each column. The general opinion is that the cost of the heavy bus loop has not been justified by experience. Electrical fixtures in solvent operation areas are of the type suitable for Class I, Group D, hazardous locations.

The entire floor area is pitched 0.125" to the foot towards floor drains which are located in the centres of each 16' x 12' rectangle except down the centres of the room. A slightly greater pitch has been found advisable to provide more convenience and comfort to the staff.

Steam condensate lines are located in pitched trenches which run along both side walls below the floor level. The trenches are covered flat with the floor level by flat steel plates with non-skid surface pattern.

The pilot-plant area is heated by means of low-pressure steam space heaters suspended below the second floor along the sides of the rooms. Ventilation is by means of 4 motor-driven exhausters to give one air change in 7 min. in the Southern Laboratory and 2 such are used in the Western Laboratory to give one air change in 15 min.

To provide maximum structural flexibility steel-grating balconies were included at the 3 elevated floor levels at the end of the pilot-plant area. In most of the laboratories these have been replaced by drained and curbed concrete balcony floors. This change has transformed the balconies into useful working spaces.

Various special service facilities have been installed in the pilot-plant areas. Though special provision is made for plants dealing with flammable solvents, it is generally agreed that a satisfactory location for such operation is a separate structure or out-of-doors. Safety equipment such as fire extinguishers, fire hose with fog nozzles, masks, fire blankets,

showers and protective clothing for workers are stressed as necessary, and proper location of these items deserve careful thought. The Eastern Laboratory has made extensive use of colour schemes for identifying pipe-lines making control valves and switches conspicuous, and marking out pedestrian aisles on the floor.

Although 120 lb. house steam supply is ample for most needs, 2 special installations have been found desirable. A small compressor is used to furnish high temperature steam to plattens of moulding presses. Odourless steam for food processing is supplied from a reboiler heated by house steam and fed with city water that is pre-heated to vigorous boiling in an open vessel.

Facilities for clean up include: portable vacuum cleaners with long handle attachments; water outlets provided with standard hose thread located at numerous places throughout the room; a booster pump supplying 16 gal. of water per min. at a pressure of 500 lb., permanently connected to a small line serving 2 or 3 places where particularly tough cleaning jobs are to be done, e.g. spray dryer; and steam-water mixing fixtures installed at a large number of places to provide hot water for washing. Safety of these fixtures for operators is assured by standard devices.

The pilot-plant areas have been used for several general types of activity and not merely for pilot-plant operations. They have been employed extensively for large-scale preparation work and for various kinds of engineering and technological research. The latter is particularly characterized, in part, by its dependence on the use of industrial types of equipment. Pilot-plant developments are undertaken only after careful analysis of the commercial prospects of a new process.

In each of the regional laboratories, from 20 to 30 employees of a professional grade, most of them trained chemical engineers, are concerned with experimental operations in the pilot-plant area. A locker and a shower room have been provided for clean up and change of clothes. It has been also found necessary to provide them with office space, outside the pilot-plant area, where they can keep their books and records and study or write without distraction.

Maintenance and emergency repairs are facilitated by keeping

small stocks of gaskets, belts, packing, nuts, screws and bolts and spare parts in chests or racks along the walls. The tools for assembling or cleaning a special piece of equipment should, if possible, be kept in a cabinet adjacent to the equipment.

A few standard chemical laboratories capable of accommodating 2 to 8 workers are located near the pilot-plant area.

It has been found that in the original design not enough general storage space was provided in these laboratories. Inactive equipment in the pilot-plant area obstructs working space, and leads to undue crowding of active equipment. It is recommended that at least 0.5 sq. ft. of proper storage area, under cover, should be provided for every sq. ft. of active pilot-plant area.

### New Oxygen Isotope

THE SIXTH OXYGEN ISOTOPE to be isolated is Oxygen 14 which has a brief life-period of 76 sec., after which it turns into an excited nitrogen isotope. Both positrons and  $\gamma$ -radiations are involved in its decay. The other known isotopes of oxygen are those having atomic weights of 15, 16, 17, 18 and 19 (*Sci. News Letter*, Dec. 11, 1948).

### Vitamin B<sub>12</sub> by Fermentation

THE ISOLATION OF VITAMIN B<sub>12</sub>, the new vitamin used in the treatment of pernicious anaemia, has been reported from the laboratories of *Merck & Co.*, Rahway, N.J. Red crystals of the vitamin were obtained in the fermenting media using the mould *Streptomyces griseus*. This discovery may soon make the production of the vitamin in commercial quantities possible (*Sci. News Letter*, Dec. 11, 1948).

### Use of Liquid Fuels

A CONFERENCE ON "MODERN Applications of Liquid Fuels" was recently organized at Birmingham by the *Institute of Petroleum* and the *Institute of Fuel*, at which modern trends in the design, operation and performance of burners, agricultural dryers, Diesel rail traction engines, the use of oil in the gas industry and the principles of combustion chamber design for gas turbines were discussed (*Nature*, 1948, 162, 951).

The suggested uses for liquid fuel in domestic heating are largely



based on the work of a special committee of the *Institute of Petroleum*. The fuels employed are liquefied butane and other gases, kerosene and Diesel oil. The development of modern domestic appliances burning kerosene was described and attention was directed to their high thermal efficiencies. Such appliances have many advantages in rural areas.

The conference was primarily concerned with the industrial application of oil fuels for which coal and petroleum may be used alternatively. In Britain coal is employed only because of its low cost, and the opinion is held that but for this advantage oil would be used in preference to coal. Oil has a higher calorific value, is easier to handle and store than coal, and is cleaner in use—qualities which account, for example, for the replacement of coal by oil for use at sea.

The oil flame has a higher radiation efficiency than the gas flame—a property which has contributed to the efficiency obtaining in the manufacture of steel in the open hearth furnace by conversion to oil-firing. The combined use of oil and coke-oven gas has given good results, and it was suggested that other combinations, including such fuels as coke breeze and powdered coal, might be used with advantage.

Another factor in the achievement of improved results with oil-firing is the greater ease of control which attends the use of oil. The use of oil in glass furnaces also gives the advantage of greater ease of control and increased rate of glass-melting, but the intense radiation from the oil flame results in increased wear on refractories and, at present, is not economical in comparison with efficient producer-gas system. The assessment of the relative merits of coal and oil-firing involves many technical and economic factors.

### New Phosphating Compositions

TWO RECENT PHOSPHATING PATENTS relate to the use of double fluorides for coating metallic surfaces which have been found difficult to coat hitherto, especially surfaces which are predominantly of zinc or zinc alloy and iron (*Chem. Age*, 1949, 60, 20). These ensure better paint adhesion to coated surfaces.

To an aqueous solution of zinc or manganese dihydrogen phosphate (0.3 per cent metal) are

added a soluble salt of nickel (nickel sulphate, 0.3 per cent nickel), a fluoborate and preferably a nitrate or nitrite ( $\text{NO}_3$ , 0.3 per cent). Immersion for about 1 min. at 150°F. or even 15 sec. is sufficient for electroplated and galvanized metal and high aluminium-zinc alloys. The same solution may be sprayed or run over the surface. The addition of fluoborate increases the etching tendency and is of special value in securing good paint adhesion on surfaces containing aluminium.

A second formula relates to phosphating of surfaces predominantly of aluminium or its alloys. Solutions containing an acid phosphate, fluoborate and an oxidizing agent as principal reagents are sprayed on to the surfaces to be coated. A replenishing solution is prepared by dissolving in water 1,295 lb. of phosphoric acid (75 per cent), 700 lb. of nitric acid (42°Be), 830 lb. of zinc oxide, 730 lb. of boric acid, 1,018 lb. of hydrofluoric acid (60 per cent) and 307 lb. of sodium fluoride.

### Coconut Oil-base Detergent

A NEW COCONUT OIL-BASE SYNTHETIC detergent called Hi-10 is made by *Eureka Chemical Corp.* at Los Angeles. The product is odourless and non-soluble. It is obtained by combining coconut oil with amines. The result is a penta-valent nitrogen compound in which coconut fatty acids are combined with amines and aminomethyl compounds.

The new detergent has been employed to remove odour from railroad refrigerator cars in which fruits and vegetables are shipped. It can also be employed for general cleaning (*Chem. Met. Eng.*, 1948, 55, 196).

### New Anti-oxidants

TWO NEW ANTI-OXIDANTS, TENOX HQ and Tenox BHA, have been developed by the *Tennessee Eastman Corp.* Added to fats and oils during processing, small percentages of the two compounds are reported to retard effectively the development of rancidity. Indications are that these compounds, used singly or in combination, will stabilize fats and oils throughout the normal time of processing, distribution and consumption. Toxicity tests have shown that the products are safe for human consumption when used in prescribed proportions. They do not

impair the nutritive value of oils or food products, and are economical for use.

Tenox HQ, a highly purified form of hydroquinone, is an effective stabilizer for salad oils, cooking oils, butter, margarine, orange and lemon oil extracts and dried milk. It prevents loss of vitamin A in oils containing it. Tenox BHA, butyl hydroxy anisole, is of special value in stabilizing animal fats, particularly lard. In combination with citric acid, it provides an adequate answer to the stabilization of lard for all uses including frying of potato chips, doughnuts and baking of crackers or pastry (*Chem. Met. Eng.*, 1948, 55, 198).

### Sugar Refining

THE COST OF REFINING SUGAR CAN be greatly reduced by a new process which makes possible the recovery of vitamins now largely wasted (*Chem. Eng.*, 1948, 55, 306). The raw sugar of 95 per cent purity can be refined to 99.7 per cent purity by washing with wood alcohol. The valuable constituents are obtained in a residual molasses of pleasant flavour, and can be directly employed to supplement human diet.

### U.S. Committee on Pacific Oceanography

THE FIRST MEETING OF THE Committee held during July 1948 was mainly concerned with American participation in the Seventh *Pacific Science Congress* to be held in New Zealand this year. Two symposia, one concerning conservation of marine resources, and the other on marine biogeographical provinces in the Pacific basin, are included in the programme. Another topic discussed at the meeting dealt with the need for standardizing gear and methods, and with methods and arrangements for correlating and integrating programmes of research in the oceanography of the North Pacific Basin.

A special report on the development of Hawaiian oceanographic research, the establishment of the Hawaii Marine Laboratory, and the initiation of a programme of research on tunas, was presented at the meeting. For the training of oceanic biologists, arrangements have been made with Rhode Island State College and its Narragansett Marine Laboratory to provide academic training and field experience for graduate

students prior to their assignment for study under specialists at Woods Hole Oceanographic Institution (*Pacific Sci.*, 1940, 2, 299).

### Pacific Fisheries Investigation

A SUM OF \$1,000,000 HAS BEEN made available to the U.S. Fish and Wildlife Service through a U.S. Congress Bill to implement what is known as "Farrington Fisheries Programme". This programme provides for the "exploration, investigation, development and maintenance of the fishing resources; development of the high seas fishing industry of the territories and island possessions of the United States in the tropical and sub-tropical Pacific Ocean and intervening seas". The Bill authorizes U.S. Fish and Wildlife Service to secure laboratories, vessels and personnel to investigate the biological, technological and economic problems connected with tunas and tuna-like fishes. Oscar E. Sette, former Chief, South Pacific Investigations of the U.S. Fish and Wildlife Service, with headquarters at Stanford University, has been appointed Director. A new laboratory and administrative headquarters will be constructed at the University of Hawaii (*Pacific Sci.*, 1948, 2, 299).

### Reflective Radiant Conditioning

A NEW SYSTEM OF MAINTAINING year-round indoor-comfort has been reported from the University of Cincinnati. The new system is called "Reflective Radiant Conditioning". Walls and ceiling surfaces are embossed with aluminium foil and the heating equipment required is a few electrical resistance coils in each room for generating heat rays. Cooling coils, resistance wires for heat and fluorescent colour lighting are all installed in an alcove near the ceiling (*Sci. News Letter*, Dec. 11, 1948).

### New Trends in Laboratory Glassware Manufacture

MANY BRITISH FIRMS, IN PARTICULAR, Messrs H. J. Elliott Ltd., Treforest Trading Estate, Glamorgan, have introduced many improvements in the character and packaging of glassware. The firm is marking their volumetric glassware "E-Mil Green Line" complying with the *National*

*Physical Laboratory*, Grade A specifications and "E-Mil Gold Line" for Grade B standard. The graduations are filled with insoluble green and yellow enamels. The pigments are somewhat fluorescent and are easily visible in poor lighting even with coloured solutions, and the markings are fused into glass.

Stoppers in graduated flasks and cylinders have often proved troublesome owing to a propensity for "seizing". The new "E-Mil" stopper is a valuable accessory. Accurately moulded from an inert plastic, it provides a gas-tight and liquid-tight closure to an orifice of the *British Standards Institution* Specification taper, but does not bind or seize up irremovably.

The firm has introduced an innovation in packaging of articles to reduce losses in handling. Each article is sealed into its own specially fitting carton, which can be retained by the purchaser for safe storage of the article. The extra expenses of carton packing will be offset by dispensing with crates, etc. (*Nature*, 1948, 162, 974).

### Plant Disease Forecasting Service

A NATIONAL PLANT DISEASE Forecasting Service has been initiated in America for the benefit of agriculturists. The warning service, which is an undertaking by the United States Department of Agriculture, 37 State agricultural colleges and 6 Canadian provinces, promises to bring farmers more money by keeping down crop losses and by preventing unnecessary expenditure on spraying and dusting for plant disease epidemics that do not develop. Disease forecasting helps to make better use of known prevention and control measures and provides timely warnings against outbreak of plant diseases.

The service was started in 1947 with a warning service on late blight of tomatoes following the disastrous outbreak of this disease in 1946. In 1948 the service was expanded to include late blight of tomatoes and potatoes, blue mould of tobacco and downy mildew of cucumber, cantaloupe and watermelon.

Disease outbreaks are reported by key scientists throughout the United States and Canada. Reports from these scientists go to the Plant Disease Forecasting Headquarters at Beltsville, Mary-

land. Emergency conditions are reported by telegram. A summary of these reports is dispatched to the Agricultural Insecticide and Fungicide Association, which in turn passes it on to manufacturers and distributors so that adequate supplies of dusting and spraying materials can be sent to danger areas and stored (*USIS*).

### Transit Instrument

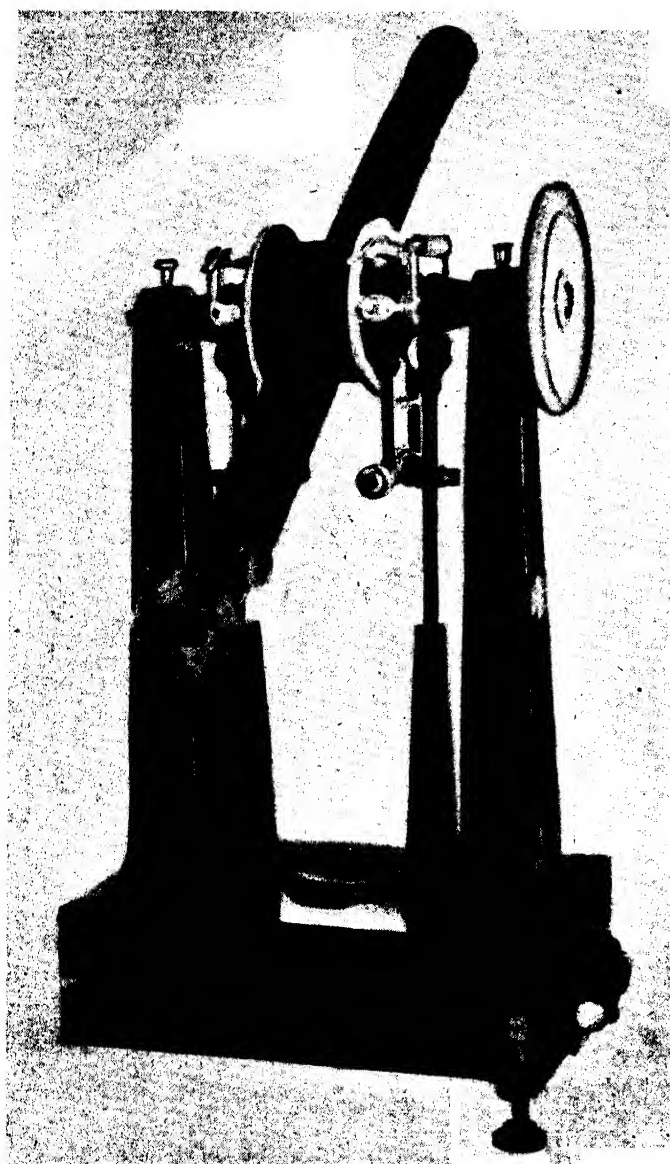
THE *Andhra Scientific Co. Ltd.* of Masulipatam has designed and fabricated a transit instrument which was exhibited at the January session of the *Indian Science Congress*. The optical system consists of a 3" or 4" apochromatic objective (triplet), precisely corrected for chromatic and spherical aberrations and coma, and a Ramsden type eyepiece. The aperture ratio is 1:15. In the field of view of the eyepiece are placed 2 gratitudes, one of which is fixed while the other can be moved by means of an accurate micrometer screw. For convenience of observation, the micrometer and eyepiece are fitted into a star diagonal prism adaptor.

Two circular scales with 2 verniers each are mounted on the horizontal axis of the instrument which is exactly perpendicular to the optical axis of the telescope. One of the circles is divided into  $\frac{1}{2}$  deg. and can be read correct to 1', while the other is divided into  $\frac{1}{4}$  deg. and can be read correct to 10". Magnifiers which are focussed by spiral motion are fixed to each vernier for taking readings conveniently. The graduations of the scales are on solid silver worked into the brass body drawn with a fine diamond point on a precision dividing engine. The verniers are of solid silver. Both the scales are given a dull finish and lacquered for protection after filling up the graduations with a black pigment. The scales are tested to a high standard of accuracy.

The micrometer consists of a slide carrying a graticule which moves relative to a fixed graticule with a central vertical line. The movable graticule has 5 vertical lines at 2 mm. intervals (lying in the centre of 2 similar lines 16 mm. apart) with 2 horizontal lines (2 mm. apart) running across them. The screw of the micrometer is of the precision type and made of low expansion steel.

The illumination for the graticule is provided by a right-





TRANSIT INSTRUMENT.

angled prism so fixed in the hollow axis of the instrument as to reflect a small pencil of light entering the axis from an outside source on to the graticule. The whole arrangement is so designed as to give only a diffuse oblique illumination. The prism can be adjusted from the outside by means of a milled knob.

The level tubes mounted on the instrument are  $\frac{1}{4}$ " in dia. and are precision ground to give the exacting sensitivity required. These are thoroughly tested to

within very close limits prior to and after mounting.

The axis of the instrument, which is hollow and of good quality brass with a central cube to take the telescope, is provided with hardened steel-bearings moving in gun metal bushes carried on 2 elegant and sturdy cast iron supports. The weight of the axis is mostly taken up by 2 spring loaded anti-friction rollers.

The heavy cast-iron base is provided with coarse and fine

adjustments, and can be clamped in any position.

The whole instrument is finished in a pleasing dull-black enamel with some brightly nickelled parts as relief, presenting an elegant picture of Indian skill and craftsmanship.

### A New Maximum & Minimum Hygrometer

A NEW TYPE OF HYGROMETER, THE "Mahajan Maximum and Minimum Hygrometer", has been developed by Dr. L. D. Mahajan, Director, Meteorological Observatory, Patiala. Just like the maximum and minimum thermometer, this instrument records the maximum and minimum humidities for any given period. The hygrometer is provided with 3 pointers indicating, in addition to maximum and minimum humidities, the percentage humidity of the surrounding atmosphere at the time of reading. It serves the purpose of a hygrograph also and saves much labour and expense. (Contributed)

### Standard Atmosphere for Testing

FOR MAKING PROPER AND EQUI-table comparisons of test data on various commodities, materials and equipment, an agreed set of atmospheric conditions under which tests may be carried out are necessary. The I.S.I. Sectional Committee on Textile Standards (TDC 1) prepared a note on the subject of *Standard Atmosphere for India* which has been published in the first number of the *Indian Standards Institution Bulletin*, a quarterly publication issued by the I.S.I. (1949, 1, 5).

No standard atmospheric conditions have been defined for any tropical country, at any rate, for one having atmospheric conditions of the type prevailing in India. This has resulted in:

(1) The non-existence of satisfactory installations of air-conditioned rooms for testing.

(2) Installations of a few testing rooms maintained at varying conditions arbitrarily fixed, and testing being carried out without any standard atmosphere.

(3) Confusion in reporting, comparing and evaluating test data.

The proposed standardization of standard atmosphere (SA) in India is based on the following principles:

(1) Temperature and relative humidity conditions prescribed

COUNTRY	CONDITIONS			
	TEMPERATURE		RELATIVE HUMIDITY	
	°F.	Tolerance °F.	%	Tolerance %
U.K.	70	2	65	1
U.S.A.	70	2	65	2
Australia	70	5	65	2
Other European countries	68	- 9	65	2
India (proposed)	80	- 3.6 3	80	2

must be suited to the majority of tests requiring SA such as tests on textiles, paints, plastics, paper, batteries, organic materials in general including biochemical and biological products, etc.

(2) The equipment required for maintaining SA must be free from complications, and should be economical in initial cost, operation and maintenance.

(3) The standard atmosphere must be comfortable for workers and operators engaged in testing.

(4) Direct and indirect relationships with the prevailing weather conditions during the different seasons of the year in different parts of the country on the one hand, and the three basic requirements mentioned above on the other, should receive due consideration in fixing the SA. For example, the closer the SA conditions are to the general weather conditions, (a) the closer will be the correspondence between laboratory test results and the actual service value of tested materials; (b) the more economical will be the equipment and installations required in respect of initial cost, operation and maintenance; and (c) the more comfortable and healthy will it be for workers who are constantly and alternately exposed to the SA and the outer atmosphere.

A comparison of the SA adopted in various countries with that proposed for India is made in the Table above.

### Aerial Survey of Nepal

THE *Air Survey Company of India Ltd.*, a subsidiary of the *British Fairley Aviation Company* which has been mapping the Indian sub-continent for the last 25 years, has undertaken an aerial survey of Nepal. The object of the survey is to complete a topographical map of 29,000 sq. miles of catchment area of the 3 main rivers of the Gurkha country. The equipment of the expedition team includes air cameras, processing machinery and mapping

apparatus weighing more than half a ton.

14 aircraft are to be used at different times from the base at Dum Dum aerodrome, Calcutta. The survey is expected to complete aerial photography in 2 flying seasons (14 months) but the ground staff of cartographers are likely to take two and half years to finish their job. Even then only 20,000 sq. miles of Nepal (out of 54,000 sq. miles) would have been covered.

Aerial survey may cost anything from £10 a sq. mile for elementary work such as recording a surface without contouring to £100 a sq. mile for complex and difficult work requiring the aid of radar and other expensive ground installations.

### Water Requirements of Crops

THE GOVERNMENT OF INDIA, ON the recommendation of the *Central Board of Irrigation*, have decided to appoint a Standing Advisory Committee for research on water requirements of crops consisting of representatives of the *Central Board of Irrigation*, Ministry of Agriculture and the Meteorological Department.

The Committee will advise on research already being conducted in regard to the most advantageous use of water for agricultural purposes and problems incidental thereto. It will study and co-ordinate research work which is being done by the *Indian Council of Agricultural Research*, Agriculture and Irrigation Departments in Provinces and States and agencies concerned with multi-purpose projects, and advise on the most satisfactory method of achieving optimum results in this field.

### Railway Research Council

THE GOVERNMENT OF INDIA HAVE set up an Advisory Research Council for Indian Railways "to improve efficiency, effect economy

and co-ordinate research on subjects connected with the railways". Indian railways are the largest consumers of coal and of ferrous and non-ferrous metals, and spend over Rs. 30 crores annually for the purchase of these requirements. The Council will function as a "screening body" and pass on such technical problems as cannot be tackled by railway administrations to research institutions within the country.

The Council consists of the following members; Mr. K. C. Bakhle, Chief Commissioner of Railways; Dr. S. S. Bhatnagar; Prof. K. S. Krishnan; Dr. D. R. Malhotra; and Mr. P. L. Verma.

### Tea Committee for India

THE GOVERNMENT OF INDIA HAVE decided to form a *Tea Committee* which will replace the existing *Indian Tea Market Expansion Board* founded in 1903. The *Committee* will consist of representatives of the tea industry, the Central Government and various tea-growing Provinces and States, and will work under the control of the Central Government. The functions of the *Committee* will be mainly to promote research, collect statistics, improve marketing conditions, and advise tea growers and exporters on all matters relating to tea.

### Jubilee Number of "Capital"

THIS BEAUTIFULLY GOT-UP souvenir number, issued by India's well-known journal of economics to mark the completion of its 60 years of publication activity, is a valuable record of permanent value, of developments in the fields of commerce, trade, industry and finance in India. Short biographies of personalities associated with the journal, India's credit structure, joint stock enterprises, insurance and other economic problems, a survey of plantation crops like tea and jute, and of paint, varnishes and salt industries form the subjects discussed in its 150 pages. The last item, "A Chronicle of 60 Years", gives a résumé of important political and economic events in India and elsewhere.

### Reward for Groundnut Hand Decorticator Announced

A PRIZE OF Rs. 2,000 WILL BE awarded by the *Indian Oilseeds Committee* to any person or body

who designs the best model of groundnut hand decorticator and demonstrates its working to the satisfaction of the *Indian Oilseeds Committee*. Entries accompanied with diagrams showing details of the machine should be submitted to the Secretary, *Indian Oilseeds Committee*, Ministry of Agriculture, New Delhi, not later than October 31, 1949. Competitors should be prepared to demonstrate the working of the machine at such place and time as directed by the *Committee*.

The groundnut hand decorticator should fulfil the following conditions: (i) the hand decorticator should, as far as possible, be made of material easily available in villages. It should preferably be made of wood and should have as few iron parts as possible; (ii) the construction should be simple so that repairs and spare parts can be provided in the villages by the village carpenter or blacksmith; (iii) the grates or sieves used in the machine should be adjustable to enable proper decortication of groundnut pods of different varieties and sizes; (iv) the turn-over and cost of decortication should compare favourably with those of power decorticators; (v) the proportion of split and broken kernels, "nooks" and unshelled pods in the decorticated produce should be insignificantly small; and (vi) the cost of the hand decorticator should not exceed Rs. 100.

### Announcements

*UNESCO Grant for Indian Chemical Society* — The *Union Internationale d' Histoire des Sciences* has, out of their subscription from UNESCO, contributed a sum of \$500 towards the preparation of "History of Chemistry in Ancient and Medieval India".

*Central Food Technological Research Institute, Mysore* — A Local Planning Committee consisting of the following members has been set up to expedite the work of organizing the Institute: Hon'ble Mr. K. C. Reddy, Prime Minister, Mysore (*Chairman*); Prof. Mata Prasad (Royal Institute of Science, Bombay); Dr. G. Shankaran (All-India Institute of Hygiene and Public Health); the Director, Indian Institute of Science, Bangalore; the Director, Council of Scientific & Industrial Research or his nominee (*Ex-officio*); a representative each of the Food Ministry and Health Ministry of the Government of India; and

Planning Officer, Central Food Technological Research Institute (*Secretary*).

*Central Drug Research Institute, Lucknow* — The appointment of a Local Planning Committee consisting of the following members has been announced: Hon'ble Shri C. B. Gupta, Minister, U.P. Government (*Chairman*); Acharya Narendra Deo; Dr. B. Sahni; Mr. A. N. Jha; the Director of Medical and Health Services, U.P.; the Principal, Medical College, Lucknow; Chief Engineer, P.W.D., Lucknow; the Executive Officer, Improvement Trust, Lucknow; a representative of the pharmaceutical industry; a representative of the Central Legislature; and Planning Officer, Central Drug Research Institute (*Secretary*).

*Second International Conference on Soil Mechanics & Foundation Engineering* — Sets of the printed proceedings (5 vols.) of the Conference held in Holland during June 1948 are available for sale. Those interested may secure copies directly from the Secretary, Organizing Committee, Stichting Waterbouwkundig Laboratorium, Delft (Holland), or may contact the Secretary, Central Board of Irrigation, Simla. The cost per set is Rs. 100.

*"British Chemicals & Their Manufacturers"* — The 1949 edition of this Directory published by the *Association of British Chemical Manufacturers* includes up-to-date information on the manufacturers of chemicals in Britain and supercedes the 1946 edition.

The Directory can be had free by any inquirer writing on business paper or giving a genuine indication of his being likely to put the publication to good use as a purchaser of chemicals.

## REPORTS FROM STATES & PROVINCES

(continued)

On the animal husbandry side, 4 regions have been proposed: (1) *Dry Belt Region* consisting of Eastern Punjab, Delhi, Rajasthan and West U.P.; (2) *Wet Region* including Bengal, Assam, Bihar, Orissa, Madras, Travancore and Cochin; (3) *The Medium Rainfall Region* consisting of Hyderabad, Central India States and C.P. and Berar; and (4) *the Himalayan Region*.

## BOMBAY

### Ship-building Yard

BOMBAY IS TO HAVE A SHIP-building yard bigger and better equipped than the Vizagapatam ship-building yard, if the proposed 5-year plan submitted by the Provincial Government to the Government of India's Ship-building Committee is adopted. The scheme estimated to cost Rs. 2.5 crores has been drawn up by the Chief Engineer of the Bombay Port Trust.

The proposed yard is to be located at Trombay, about 15 miles north-east of Bombay, and 10 ship-ways are planned so that the construction of 10 vessels up to 15,000 tons capacity could proceed at the same time. Room for further expansion and fitting of 2 berths is provided for in the plan.

The Trombay site is eminently suitable owing to its ample sea frontage for launching vessels and a vast area of land in the rear. All labour required can be had from the adjacent maritime districts.

### Manufacture of Penicillin

THE GOVERNMENT OF INDIA HAVE decided to set up a plant to manufacture penicillin in Bombay. The work is to commence in a couple of months. The plant, which is expected to be complete in 2 years, will cost about 2 crores of rupees. It is proposed to manufacture 100,000 million units of penicillin in the initial stages and to gradually increase the output 4 times. Production of large quantities of anti-malarial and sulpha drugs are also included in the programme.

## MADRAS

### Livestock Improvement

THE GOVERNMENT HAVE OPENED 2 new veterinary institutions at Arkonam and Pithapuram bringing the total of such institutions in the province to 138.

A scheme for the prevention of contagious diseases spreading from adjacent provinces is under consideration. Sanction has been accorded for running a breeding-tum-dairy farm at Vizagapatam. A beginning has been made for breeding the fine wool sheep of the Nilgiris, and necessary lands have been allotted on the Wenlock downs on the Nilgiris. A combined sheep and cattle farm is being established at Kurikuppi in the Bellary district.

# Reports from States & Provinces

## MYSORE

### Gold Prospecting

MYSORE'S BIGGEST IRRIGATION project — the Lakkavalli reservoir on river Bhadra — will submerge extensive tracts of lands with gold-bearing quartz veins. It is, therefore, proposed to recover all the gold before this 10-year irrigation project is put through. Exploratory work at Bellara has disclosed that the extent of the reef is nearly twice the original estimate. A 10-year scheme was approved in 1947 to develop these areas. To meet the increased power requirements for the development programme, the Government have recently sanctioned a scheme at an estimated cost of Rs. 2.46 lakhs to construct a dam across the Suvarnamukhi river. Meanwhile steps are being taken to equip the Bellara mines with modern milling and cyanidation plants.

## NEW DELHI

### Reorganization of Agricultural Research

THE ADVISORY BOARD OF THE *Indian Council of Agricultural Research* at its annual meeting held in New Delhi during January approved a number of research schemes including those on improvement of crops, agronomical conditions in general, soil conservation in Himachal Pradesh, setting up a suitable organization for collection of agricultural statistics, research on tuber crops as a substitute for cereals, and a standard method for milk analysis and dairy development. Particular emphasis was laid on research schemes in East Punjab and West Bengal for agricultural rehabilitation.

In the absence of reliable data on crop yields, the results of Government development plans on the Grow More Food and other schemes cannot be gauged properly, and the Board felt that an organization was required to collect accurate statistics. Accordingly, the Board has recom-

mended that the random sampling method should be adopted in the 5-year co-ordinated scheme for conducting surveys on the principal crops of India. The main objects of the scheme are : to evolve a random sampling technique for crop-cutting experiments under the auspices of the Provincial Agricultural Departments, to demonstrate the feasibility of the method devised, to train the provincial staff in the technique, to estimate the yield per acre for the province as a whole and to revise the present state of district normal yields.

The total expenditure involved in the scheme is estimated to be Rs. 59 lakhs.

A soil conservation scheme of Himachal Pradesh including ecological survey, cultivation of soil binders and collection of erosion data was recommended. A scheme for the improvement of soil conditions in the spent-up orchard land in the Kumaon Hills was sanctioned. A scheme to check soil erosion and the spreading of the desert area in Jaipur was approved.

The Council had at its previous meetings recommended the cultivation of tubers such as sweet potatoes and casava, and had invited Provincial Governments to submit schemes on the subject. The schemes submitted by Bombay, United Provinces, East Punjab, Bihar, Orissa and Mysore were scrutinized and approved.

Schemes for the improvement of rice crops in East Punjab, Coorg and West Bengal, and of maize in West Bengal and Bihar were sanctioned. Several research schemes on the improvement of pulses in Madras, West Bengal, U.P., Bihar, Hyderabad, Orissa, Mysore and Gwalior as also research schemes on the improvement of millets in Mysore and palmyra in Madras were approved.

A scheme for the co-ordination of research on insect pests and on the blast and foot diseases of rice in Madras, and on wilt disease of gram in E. Punjab were sanctioned. The Council recommended that the animal nutri-

tion scheme for Madras which was stopped in 1943 should be revived.

A scheme to explore the possibilities of using milch cows for draught work was considered and approved. It was suggested that the work should be conducted at 4 centres including the Indian Dairy Research Institute, Bangalore. The standardization of methods of analysis of milk and milk products, suggested in a scheme of the Indian Dairy Research Institute, was sanctioned.

Other new schemes considered and approved by the Board were : cattle-breeding in Rajputana, research on insecticides in East Punjab and cultivation of medicinal plants in Himachal Pradesh.

### Regionalization of Agricultural Research

AT A MEETING OF THE REGIONALIZATION Committee of the *Indian Council of Agricultural Research* held in January, research on the basis of different soil and climate categories was decided upon with a view to ensure planned agricultural development in all parts of the country. It was tentatively decided to divide the country for research purposes into different regions and to set up Regional Committees.

The following 5 regions were suggested both from the point of view of research on principal crops as well as on the requirements of animal husbandry :

(1) *Dry Northern Region* comprising of East Punjab, Western U.P. and Berar and parts of Rajasthan, where wheat is the chief crop.

(2) *Eastern Region* comprising of Assam, Bengal, Bihar, Orissa, East C.P., East U.P. and East Madras, where rice is the chief crop.

(3) *Southern Region* — Jhansi in south United Provinces, C.P. and Berar, Central India States, Western Hyderabad, Deccan, Western Madras, Eastern Bombay, Baroda and part of Mysore, where millets are the important crops.

(4) *Coastal Region* comprising of parts of Bombay, Madras, west of Western Ghats, Mysore, Coorg, Travancore and Cochin.

(5) *Temperate Himalayan Region* including Assam, Sikkim, Nepal, Bhutan, Kumaon, Gharwal, Simla hills, Kulu, Chamba and Kashmir State.

(continued on page 160)

# INDIAN PATENTS

The following is a list of a few of the Patent Applications accepted in the *Gazette of India*, Part II, Section I, for February 1949.

## Inorganic Chemicals

31457. BOAKE, ROBERTS & CO. LTD.: Production of aluminium salts of organic acids: *Reacting an alkali, an aluminium salt, an organic acid, and a hydrophyllic colloid.*
37640. NATIONAL SMELTING CO. LTD.: Removal of sulphur compounds from gases: *Contacting the gases with alkali metal or alkaline earth metal oxides or hydroxides on a large surface porous support between 100° to 850°C.*
40069. GLAXO LAB. LTD.: Preparation of salts of penicillin: *Penicillin dissolved in a solvent is treated with a salt of primary organic base and an organic acid.*
40070. GLAXO LAB. LTD.: Preparation of the ammonium salt of penicillin: *Ammonium salt of penicillin is treated with an ammonium salt of organic acid.*
39432. NATIONAL RADIO & ENGINEERING CO. LTD.: Electrical moulding composition: *Consisting of shellac, resin, alkaline earth salt of resin and a filler.*
38944. SEAILLES: Method for making a water solution of raw aluminates of lime and its application to the manufacture of alumina: *Dissolving lime aluminate in aqueous solvent containing alkali in dilute condition.*
39249. SEAILLES: Method for making a water solution of raw aluminates of lime and its application to the manufacture of alumina: *Adjusting the proportions of calcium aluminate and alkaline lyes so that a mixture of alkali and calcium aluminate is formed.*
39687. UNITED STATES RUBBER CO.: Recovery of fluosulfonic acid (Addition to No. 39686): *Admixing fluosulfonic acid containing free sulphur trioxide with spent acid, distilling the resulting mixture.*
40259. WILLIAMS: Process for converting sulphur dioxide to hydrogen sulphide: *Passing a mixture of sulphur dioxide, oxygen and steam over incandescent carbon.*

## Organic Chemistry

38574. DISTILLATION PRODUCTS INC.: Process of separating delta-tocopherol from vegetable oils: *Treating vegetable oil to obtain mixed tocopherol concentrate and then separating delta-tocopherol by fractional crystallization or chromatography.*
38764. ELLIOTT & AUSTRALIAN DRUG PTY.: Extraction and recovery of alkaloids: *Extracting with acid or neutral aqueous or aqueous alcoholic solution, treating the solution with water immiscible phenol and recovering alkaloid from the phenol phase.*

## Analytical Chemistry

38784. AMERICAN CYANAMID CO.: Purification of sugar solutions: *Treating sugar with acid and alkaline contact reagents and regenerating it with hot regenerating agent.*

## Medical Research & Practice (INCLUDING CLINICAL APPLICATION OF DRUGS & PHARMACEUTICALS)

38761. ELI LILLY & CO.: Crystallization of penicillin: *Precipitating penicillin salt with acetone containing up to 4 per cent water, dissolving the precipitate in acetone containing up to 10 per cent water and reducing the amount of water.*

## Metals & Metal Products

40497. BRITISH NON-FERROUS METAL RES. ASSOC.: Production of metal coatings upon metals: *Dipping metal article into liquid lead-containing nickel, cobalt manganese or chromium.*
39938. TISSOT-DAGNETTE: Aluminium solder and a method for the production thereof: *Comprising of an aluminium-containing metal and a silver-containing metal.*

## Paper & Allied Products

40456. COLUMBIA RIBBON & CARBON MANF. CO. LTD.: Carbon papers: *Backing the carbon paper with an oil repellant coating.*

## Photographic & Optical Goods

39445. MUDALIAR: Manufacture of watch glasses: *Disc cut from scrap glass pieces or electric bulbs placed in metal dies, heated in furnace and trimming edges on grind stone.*

## Rubber & Rubber Products

40722. WINGFOOT CORP.: Frothed latex sponge rubber: *Containing frothed rubber latex sponge particles of at least 32 mesh.*

## Textile & Textile Products

38010. I.C.I. LTD.: Manufacture and application of new textile treatment agents: *Mixing polycarbonate with formaldehyde.*
36795. SYREENI: Method of producing colour mixtures: *Mixing dyestuffs or colouring materials according to a classification.*
37868. INDIAN JUTE MILLS ASSOC. RES. INST.: Preparation of a fast-to-light substantially white jute: *Subjecting bleached jute to an acetylation treatment.*
38260. I.C.I. LTD.: Improvements in colouring processes: *Dyeing in the presence of benzyl alcohol.*

## Miscellaneous

33995. CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE: Production of nuclear energy: *Comprises a mass of uranium or thorium having helium or deuterium distributed therein to change the velocity of neutrons below the range of resonance velocities.*
34000. CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE: Apparatus for the production of energy by nuclear fission: *During bombardment of uranium with neutrons the unlimited reaction chain is allowed to develop and checked periodically.*

## Stabilization of Plant Names

“INTENDING to write a semi-popular book about the common trees of Malaya, I imagined that their botanical names would have been fixed. But I soon discovered that this was far from being the case. Indeed it was not uncommon to find that what was apparently the same tree was referred to by different names by each of several different authors. Thus, when Hooker, Trimen, Valetton, Merrill, and other authors of standard systematic works concerning the flora of Malay Peninsula and adjoining territories showed little uniformity in nomenclature, it was not surprising to find the nomenclature of such standard economic works, such as those of Watt, Heyne and Burkill, to say the least, unreliable... For my own satisfaction, I have adopted the principle that no alteration in botanical names should be accepted unless supported by botanical proof by which means only fact, rather than opinion, becomes the basis of argument.”

So wrote Dr. E. J. H. Corner\* in 1939. The difficulties which confronted Dr. Corner are familiar to all those concerned with the study of plant life, and arise from the single fact that the *same* plant is often referred to by different authors by different scientific names in different publications. Even a cursory reading of journals devoted to systematic botany would be sufficient to show how changes in plant names are being constantly made. This is perhaps inevitable in a science like botany which is not an exact science in the sense that mathematics, for example, is. Researches constantly bring out fresh evidence for the validity or otherwise of names, and lead to clearer understanding of the relationships among plants. Unfortunately, however, systematists are far too few and the number of plants far too large, so much so that considerable time and

labour will be required for locating all the errors and mis-applications perpetuated in literature and rectifying them. In the meanwhile, the difficulty, to which Dr. Corner has given such eloquent expression, continues to confront and confuse those concerned with the use of names for designating plant species.

Too often we find that a name published after an apparently thorough search has been changed a few years — sometimes even a few months — later. There have been numerous cases in which the name of a plant has undergone twenty or more alterations. There are instances where a name considered invalid for years has been revived, shown to be valid, and adopted. *Setaria geniculata* (Linn.) Beauv. has 73 synonyms, *Koeleria cristata* (L.) Pers. has 41, and *Echinochloa crusgallii* (L.) Beauv. has 46. Arber,\* a recognized authority on grasses, who examined the botanical names of the pearl millet, says: “The history of names which have been applied to it (pearl millet) forms an ironical commentary on the botanist’s claim that the use of a Latin terminology results in precision of meaning. This plant, early in the nineteenth century, possessed almost as many Latin names as there are floras. It has been called *Pennisetum typhoideum*, *P. alopecuroides*, *P. americanum*, *Penicillaria spicata* and *Panicum spicatum*. Of these *Pennisetum typhoideum* Pers. has been the most popular in recent years.”\* This name has since been displaced by *Pennisetum typhoides*, and, in more recent years, *Pennisetum glaucum* has been suggested as the correct name for it. Instances like this are indicative of the confusion involved in designating plants and the imperative necessity for arriving at a definite understanding on the selection and use of plant names.

\**Gardens’ Bulletin*, Straits Settlement, 1939, 10, 2.

\**The Graminae*, 1934, p. 24.

Differences in the interpretation of rules for naming plants have added to the confusion, and even in cases where only nomenclatural, not taxonomic, issues are concerned, different names have been considered valid by different botanists for one and the same plant. However, nomenclatural changes are governed, at least to some extent, by international rules, but changes based on taxonomic considerations are beyond the scope of any rules that can be framed. They depend, to a large extent, on the judgement of individual botanists. A valid name of one author may be the invalid name of another, and it is impossible to reconcile them. *Caesalpinia pulcherrima* (L.) Swartz and *Poinciana pulcherrima* L. are names of one and the same plant. So are *Chloranthus brachystachys* Bl. and *C. glaber* (Thunb.) Makino; *Cleome icosandra* L. and *Polanisia icosandra* L.; and *Brassica alba* Boiss. *B. hirta* Moench. and *Sinapsis alba* L.

There are other factors which add to the confusion in names. *Cassia obtusifolia* L. has been considered by some to be synonymous with *C. tora* L., while others consider the two to be entirely different plants. This confusion is further heightened by the suggestion that the correct name for *Cassia tora* is *Emolista tora* (L.) Britton & Rose. Similarly, *Cerbera odollam* Gaertn. is considered by some to be synonymous with *C. manghas* L., and others hold that the two are entirely different plants. When authorities differ, whose is the opinion to be accepted by a person who "uses names as appellations for plants and not as botanical footballs"?\*

### Is the Confusion Deepening?

Discrepancies are not uncommon even in the citation of authorities for plant names. Thus *Calotropis gigantea* is variously cited as *Calotropis gigantea* R. Br., *Calotropis gigantea* (Willd.) R. Br., *Calotropis gigantea* (Willd.) Dryand, *Calotropis gigantea* (Dryand) Ait., *Calotropis gigantea* (Linn.) Dryand, and *Calotropis gigantea* Ait. There can be one and only one correct author for this plant name, or for that matter, for any other plant name. All other citations must be wrong.

Protests against too many and too frequent changes in plant names have been made from time to time by botanists and non-botanists alike. Such changes are all the more exasperating when they concern

economic plants. Stabilization of plant names is the aim of systematic botanists, but they proceed to achieve this end by discovering the earliest valid name for each plant. Unfortunately, however, names considered "earliest" at one time have often been found to have earlier valid names, the discovery of which invalidates the existing names although they may have been in use for years. A systematic and rigid application of international rules of botanical nomenclature will result *ultimately* in the stabilization of plant names, but only in those cases where taxonomy is not involved. But even for this, one has to await patiently the results of extensive researches by large teams of workers well provided with ample laboratory, library and herbarium facilities. When we come to the taxonomic aspect, the position is even more discouraging. Studies on the taxonomy of economic plants are in an amazingly backward state. The literature on many genera is confused. Who is to be accepted as authorities when monographers and specialists differ? Errors in a monograph can be discovered only by another subsequent and exhaustive monographic research. Usually they have a tendency to persist and perpetuate themselves!!

### Can Usage be Ignored?

The great inconvenience caused by nomenclatural changes, at least so far as economic plants are concerned, has been well recognized and voiced by many botanists. Thus Bailey wrote: "Botanical names should not be changed lightly for the purpose of regularizing any particular scheme or plan or to make them always conform to an arbitrary set of rules. Botanical names do not belong to botanists to do with them as they will. The public has good rights in these names, and this is particularly true in the names of cultivated plants, for they may then have standardized commercial value. The only stability, of course, is usage, and usage can rarely be forced into hard and fast regulations."\* The insistence on usage and the clear recognition of the purpose served by the names given to plants are noteworthy. Chamberlain, the well-known authority on gymnosperms, is reported to have stated: "We believe that when a name has been established in literature, it should be retained... Taxonomists

\*GLEASON: *Phytologia*, 1947, 2, 202.

\**Standard Cyclopaedia of Horticulture*, 1947, 1, xv.



would do well to let familiar, well-established names alone, and not try to revive some forgotten name."\* The efforts of botanists should be diverted to the elimination of confusion due to multiplicity and instability of plant names. As Gleason has said: "If botanists search as assiduously for reasons to maintain a name as they do for reasons to change one, a considerable number of well-known names will be saved."†

Pleas for *nomina specifica conservanda* brought before the International Botanical Congress have not been hitherto accepted. At the last session of the Congress (1935), the proposal was defeated by a majority of 147 votes. It is almost certain that specific names will never be officially conserved. What is feasible, however, is the preparation of an agreed list which all botanists should accept as valid for a specified number of years, and the International Botanical Congress should be accepted as the only authority

for introducing changes in plant names. This would minimize confusion and prevent the introduction of hasty changes in names by individual workers. There has been no limit to such changes, and many controversies, often bitter, have centred round the validity of suggested or existing plant names. Confusion can be minimized if authors, who find the need for revising plant names, first obtain the approval and support of an international body of botanists. A resolution passed at the last session (1935) of the International Botanical Congress envisaged the appointment of a Committee to draw up a list of names of economic plants according to international rules, which should remain in use for ten years. This is a step in the right direction. Unfortunately, that list has not been published so far. We trust that the matter would be taken up at the forthcoming session of the Congress, and that effective steps would be taken to introduce reasonable stability in so fundamental a requirement as a name for a plant.

\* *Chronica Botanica*, 1943, 7, 439.

† *The preservation of well-known binomials* — *Phytologia*, 1947, 2, 203.

V. S. RAO

## Characteristics of the Ionosphere over Calcutta (February 1949)

S. S. BARAL, S. N. GHOSH, R. K. MITRA,  
D. C. CHOUDHURY & (Miss) T. K. PAL

*Wireless Laboratory, University College of Science, Calcutta*

THE following are the ionospheric data observed at Calcutta for the month of February 1949. The observations were made at each hour of the day for 5 days a week.

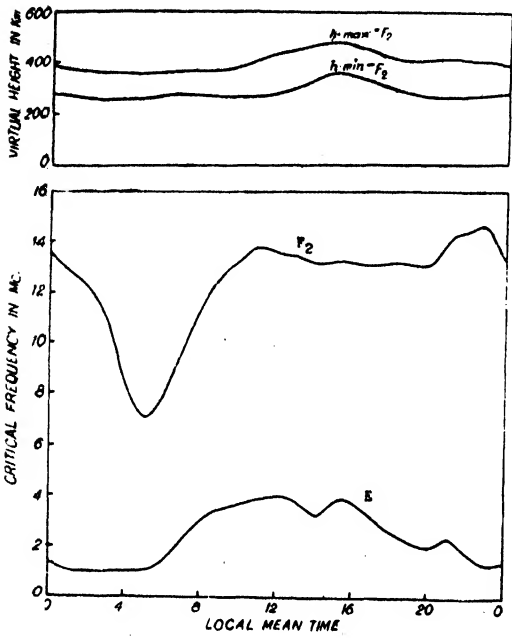
Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequencies of the E layer in graphical form during the month of February 1949. The mean of both normal and abnormal values is presented.

During this month there was high sunspot activity, and day to day observations show that on almost every day the F region

attained great heights and thicknesses during daylight hours and high ionization densities during the corresponding heights. The abnormal E ionization was also high during the afternoon and night hours.

Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during May 1949 by reflection at the F region over Calcutta, based on the normal values of the data for February 1949. Table I gives occasions when the E-region ionization was found to be abnormal and the corresponding penetration frequencies and heights.





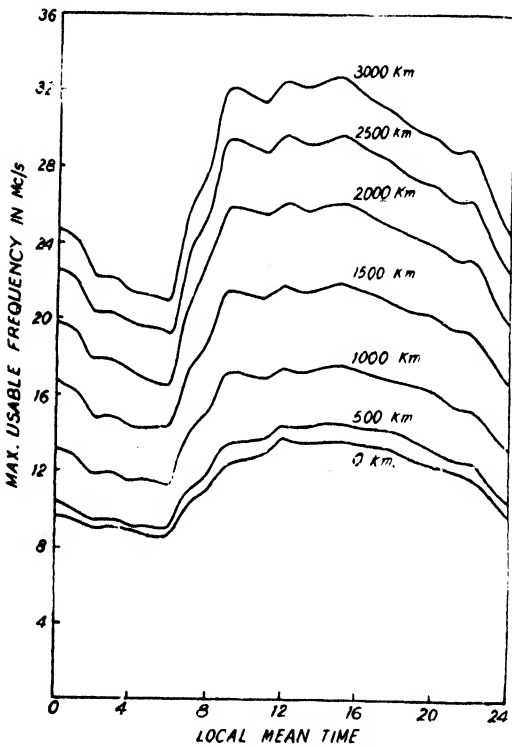
( 5 HOURS 54 MINUTES AHEAD OF G.M.T.  
FIG. 1 — FEBRUARY 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ} E_s$	$h E_s$
			Mc.	Km.
Feb. 1949	11	14.00	4.75	120
		18.00	3.30	120
		17.00	5.00	135
	16	18.00	6.60	135
		21.00	4.85	120
		22.00	4.90	120
		00.00	3.20	105
	18	01.00	3.20	105
		02.00	3.45	105
		03.00	3.55	105
		04.00	3.20	105
	19	14.00	4.70	120
		21.00	3.00	120
		22.00	3.00	120
		23.00	3.50	120
	20	00.00	4.50	120
		01.00	4.70	120
		02.00	4.65	120
		16.00	4.70	135
		17.00	4.70	135
		23.00	3.20	105

TABLE I — contd.

MONTH & YEAR	DATE	HOUR	$f^{\circ} E_s$	$h E_s$
			Mc.	Km.
Feb. 1949	21	13.00	4.80	120
		14.00	4.80	120
	22	05.00	4.25	90
		06.00	3.00	90
	23	18.00	3.60	120
		19.00	3.50	120
	24	22.00	3.60	105
		23.00	3.50	105
	25	00.00	3.25	90
		08.00	3.50	105



AT POINT OF REFLECTION.

FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION  
VIA  $F_2$  LAYER, MAY 1949.

# The Prospects of Salt Raising in the Coastal Regions of West Bengal & Orissa

JITENDRA KUMAR NAG

*Special Officer (Salt), West Bengal*

THE object of this investigation was to ascertain the prospects of salt raising along the coast-line of West Bengal and Orissa by comparing the climatic factors of the littoral of these two provinces with those of Bombay and Madras, where the salt industry has developed on a wide scale. The possibilities of reviving salt culture in these provinces have been examined by salt experts from time to time since World War I, but no serious attempt has been made so far to implement the recommendations of the experts who found in West Bengal suitable sites for establishing the salt industry.

The first inquiry (non-official) was undertaken in 1918 by the late Mr. Kapilram Vakil of Bombay, on behalf of *Messrs Tata & Sons*. Mr. Vakil's investigations were confined to the regions lying to the north of Chilka Lake, which he found to be unfavourable for the economic production of salt. The suitability of the regions near

and around the Chilka Lake for salt production had been examined more recently by Mr. F. W. Gooch, who was appointed by *Messrs Kilburn & Co.* to survey the coast-land of Bengal and Orissa. The results of this survey have not been published so far.

There is no record of official investigations undertaken during 1918-24. The Central Board of Revenue, however, put forward the view: "The possibility of manufacturing salt (in Bengal) on a commercial scale has been examined by the Government on many occasions only to be rejected. It is rendered impracticable by the low density of the sea water due to enormous discharge of fresh water from the great rivers, the prevalence of damp, cloudy weather, the scarcity of fuel for one method of manufacture (*pānga*) which in any case could hardly yield an output on any magnitude, and the occurrence of storms at critical seasons."

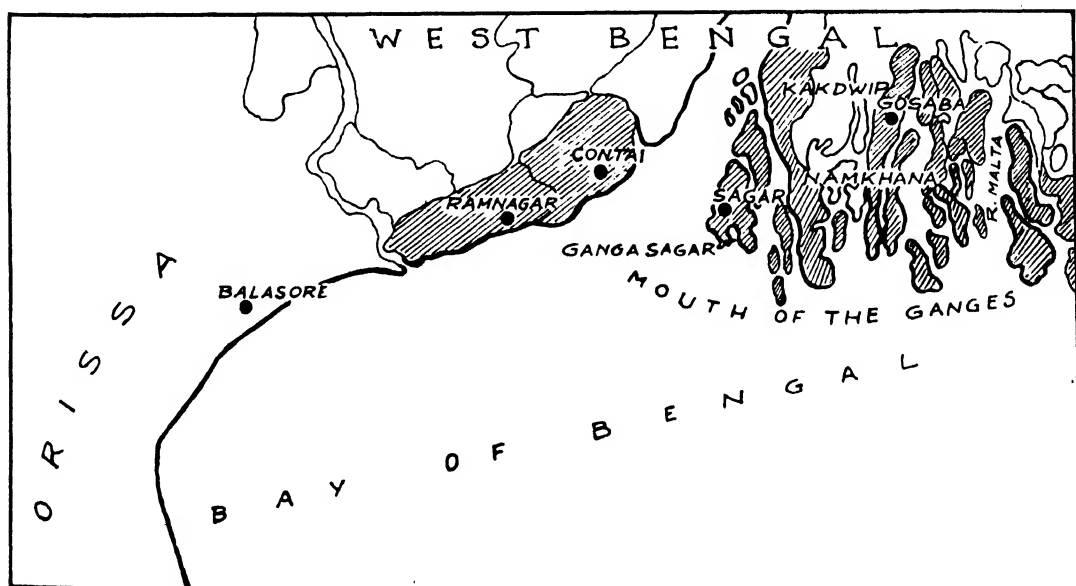


FIG. 1 — MAP SHOWING SUITABLE SITES (SHADED) FOR SALT RAISING ON THE COASTAL REGIONS OF ORISSA AND WEST BENGAL.

TABLE I — ANALYSIS OF BRINE

	DENSITY °Be.	SODIUM CHLORIDE %	MAG. CHLORIDE %	MAG. SULPHATE %	CAL. SULPHATE %	CAL. CARBONATE %
<i>West Bengal</i>						
Contai	2.7	2.2	0.24	0.10	0.1	0.01
Sunderbans	2.8	2.2	0.27	0.10	0.1	0.01
<i>Orissa</i>						
Balasore	3.0	2.4	0.20	0.20	0.1	0.01
<i>Madras</i>	2.9	2.6	0.28	0.18	0.13	...
<i>Mediterranean Sea</i>	3.0	2.7	0.61	0.7	0.04	0.02

TABLE II — AVERAGE R.H. ( 1940-44 )

PLACE	MONTHS											
	SALT SEASON											
	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sep.	Oct.	Nov.	Dec.
Bombay	76	74	77	75	76	82	87	86	86	82	72	70
Sagar Island ( West Bengal )	62	61	73	80	79	79	84	83	80	75	66	63
Madras	82	83	82	77	69	64	67	73	76	84	88	89
Vizagapatam	77	80	78	75	74	76	79	77	82	78	70	73
Balasore ( Orissa )	75	72	70	72	73	80	83	85	81	81	74	73

It may be mentioned here that the salinity of the bay water in different parts of the coast of Bengal had not been properly tested till 1937, in which year 2 officers of the provincial Government recorded the density of the sea water in the estuaries of the Sunderbans. Six years earlier Mr. C. H. Pitt of *Khewra Salt Mine* examined the extensive saliferous coast-land of Bengal on behalf of the Salt Industry Committee, but he visited only Midnapore and Western Sunderbans, leaving out the entire East Bengal seashore region. He was of the opinion that salt raising was not impossible, and suggested the starting of experimental units by private enterprise.\*

In 1937 Mr. D. N. Mukerji, now Salt Controller, Government of India, and Mr. V. S. Rao of the Bengal Forest Service undertook an enquiry into the possibilities of developing the salt industry in the Sunderbans area. They stated: "We do not agree with Mr. Pitt that the salinity of sea brine on the Bengal coast never exceeds 1.5°Bé. Mr. Pitt did not visit the Sunderbans to the east of Lothian Island and had therefore no opportunity to study the salinity of the water beyond Lothian Island."

#### Factors Affecting Salt Production

The raising of salt by solar evaporation is influenced by five factors, viz. : (i) salinity

of brine ; (ii) relative humidity ; (iii) rainfall and number of rainy days ; (iv) maximum and minimum temperatures ; and (v) wind velocity.

(i) *Salinity* — During the usual season of salt manufacture — December to May — the density of the bay water of Bengal averages to about 2.5°Bé., being almost equal in strength to that employed in the salterns of Madras and Bombay.

It is evident from the analyses that the strength of the sea water of West Bengal is a little less than that in Madras and in the Mediterranean, but not so low as to materially affect salt manufacture. Weak brine evaporates quickly in the initial stages, and does not render salt manufacture difficult or unremunerative.

(ii) *Relative Humidity* — The relative humidity influences the rate of evaporation of the brine. It has been observed, however, that when the temperature is high, and the sun and dry winds prevail, a humidity of 70-75 per cent does not greatly hinder salt raising.

The following is a comparative study of the average humidity for 5 years in places where salt is or can be manufactured on a big scale.

The figures indicate that the relative humidity of the Bengal delta is almost similar to that of the coastal part of eastern or western India. During the dry months of December to March, the West Bengal littoral is more dry than Bombay or

\* Report on the investigations into the possibilities of salt production in Bengal and Orissa, 1932.

Madras coast. The average relative humidity for 5 years for the period December to June is 71 per cent in Sagar Island (West Bengal coast-land), and 76 in Bombay. In tropical climates a period of dry weather prevails only for a short while during the winter months, and the atmosphere is moist during the rest of the year. The lands situated beyond 24°N. are arid and afford a suitable climate for sea salt culture and for that reason Karachi and Okha have developed their salt industry extensively. But moist localities like Bombay and Madras, as a whole, produce more salt than the out-turn of Karachi and Okha works taken together.

(iii) *Rainfall & Rainy Days* — In the manufacture of salt, the initial brine from sea or salt lake is concentrated only by solar evaporation till it attains a density approaching its saturation point. The concentrated saline is either led into solar pans (crystallizing beds) or into boiling pans for the deposition of salt crystals. Which ever method is adopted, a rain-free period is essential for the smooth working of a salt factory.

Rains not only dilute saline but also arrest evaporation. For this reason the salt season closes before the monsoon breaks out. The *intensity* of rainfall during the monsoon months does not affect salt manufacture as the *duration* of the rainy period. The factor for consideration in regions with heavy annual rainfall is the distribution of rainfall during the salt season irrespective of the intensity during the off season.

The rainy season in West Bengal and Orissa, as in Bombay and Madras, covers about 4 months. In Bengal as well as in Bombay it begins in June and is almost over by September. In Madras the season starts later and continues up to December. Salt raising generally starts towards the end of January and closes before June, but in Madras the salt pans may be worked up to August or even September. Occasional showers do not greatly hinder the sequence of salt culture if they are followed by clear days and sunshine. Table III gives the rainfall of West Bengal and Orissa coast-line during November to August.

The normal rainfall in Contai or Gosaba during the salt season is less than the normal of Gopalpur (Naupada) and Cocanada. The normal rainfall of Madras during the non-monsoon months, that is from January to August, is 14" and the average number of

TABLE III — AVERAGE RAINFALL IN INCHES & NUMBER OF RAINY DAYS (1940-44)

January to May are the months during which salt is actually manufactured in Naupada (Gopalpur), Cocanada, Bombay, Contai and Sagar of West Bengal. In Madras salt is raised from February till the season ends in August or September with the advent of the rains.

	NOVEMBER		DECEMBER		JANUARY		FEBRUARY		MARCH		APRIL		MAY		JUNE		JULY		AUGUST		NON-MONSOON MONTHS		ANNUAL	
	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days	Fall	Days
Gopalpur	3.9	2.4	0.5	1.0	0.3	0.6	0.9	1.0	0.6	3.0	0.7	1.8	2.1	3.2	5.5	7.4	7.2	...	7.7	...	9.1	13.0	45.0	...
Cocanada	4.9	...	0.4	...	0.2	...	0.1	...	0.9	...	1.1	...	1.4	...	4.3	...	6.1	...	3.2	...	8.9	...	41.0	...
Madras	...	...	5.8	5.2	1.4	1.7	0.3	0.6	0.2	0.3	0.5	0.7	1.1	1.1	1.9	3.7	3.9	7.0	4.6	7.9	14.0	23.0	50.8	57.3
Bombay	0.1	...	0.1	...	0.8	...	0.1	...	0.1	...	...	...	1.2	...	25 app.	...	30 app.	...	...	...	2.4	...	71.0	...
Contai	1.1	1.6	0.1	0.4	1.0	1.5	0.7	1.2	0.8	2.9	1.4	2.9	3.8	6.0	9.4	13.4	...	...	...	...	8.8	16.5	62.6	90.8
Rannagar	1.7	...	0.1	...	1.1	...	0.8	...	1.5	...	1.1	...	2.3	...	7.6	...	...	...	...	...	8.7	...	55.9	...
Sagar	1.5	1.4	0.3	0.5	0.3	0.9	1.1	1.8	1.4	2.0	1.1	2.1	4.4	5.8	11.5	12.3	14.6	15.8	14.2	16.5	10.1	14.5	69.4	79.6
Gosaba	0.7	...	...	...	0.4	0.9	0.9	...	0.9	...	1.5	...	2.8	...	10.3	...	15.3	...	1.7	...	7.2	...	61.7	...
Balasore	1.5	1.5	0.2	0.3	0.5	1.0	1.4	2.4	1.8	2.9	1.7	3.5	4.6	5.9	9.2	11.1	...	...	...	...	11.8	17.5	62.0	76.0

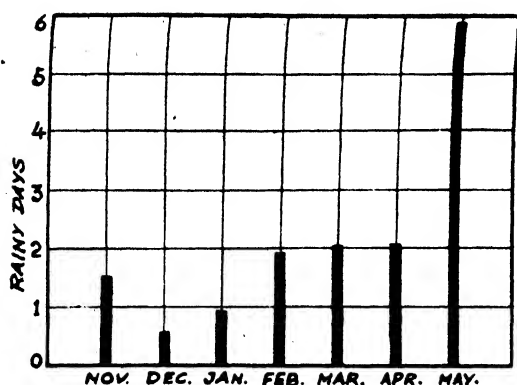


FIG. 2 — SAGAR ISLAND (WEST BENGAL) :  
NUMBER OF RAINY DAYS ; AVERAGE FOR  
50 yrs. ( 1871-1920 ).

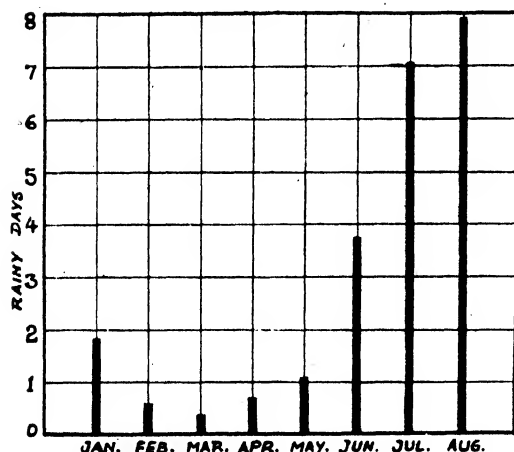


FIG. 3 — MADRAS : NUMBER OF RAINY DAYS ;  
AVERAGE FOR 50 yrs. ( 1871-1920 ).

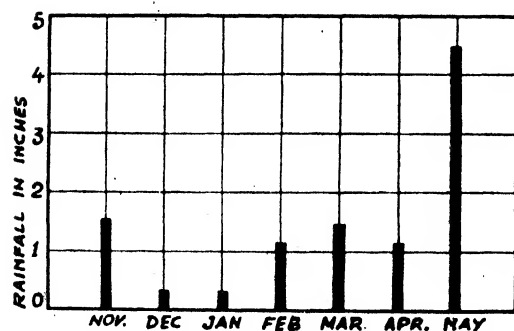


FIG. 4 — SAGAR ISLAND (WEST BENGAL) : MEAN  
RAINFALL IN INCHES ; AVERAGE FOR 50 yrs.  
( 1871-1920 ).

rainy days is 23, and yet salt is manufactured there. Gopalpur, near which Naupada produces about 18 lakh md. of salt per year — being the second productive centre of Madras province — has a rainfall of 9" and an average of 13 rainy days during the salt season due to the thrust of Norwesters. Contai is similarly subject to rain showers during the months of November to May. This is followed by 15 bright sunny days during the month of June, and if the damage done by the Norwesters be not irreparable, the salt operations are not hampered.

Balasore in northern Orissa has an average rainfall of 11.8" distributed over 17.5 days during November to May. Balasore shore is only 50 miles down Contai and during the months when salt deposits in solar pans — February to May — the number of rainy days is 14.

It has often been said that the rainfall in Orissa and Bengal is a great impediment for the successful manufacture of salt. This is incorrect. It should be remembered that Madras with frequent showers has successfully developed her salt industry and is now the prime producer of salt in the Union. The salt season in Bengal and Orissa will be shorter than that on the North Coromandal coast, but the prospects for reviving the salt industry in these provinces are indeed bright.

(iv) *Temperature* — The diurnal temperature, greatly influencing the relative humidity and the vapour pressure of the air, is an important factor in considering the amount of evaporation taking place in the solar pans of a salt factory. The temperature of the atmosphere at a given place depends on: (a) latitude; (b) elevation; (c) distance from the sea; (d) character of the wind; and (e) the amount of rainfall. During December to June the temperature of the air in India generally decreases with the increase of latitude and hence, from south to north, the isotherms run across India nearly parallel to the latitude. The climate is moderately dry. During the less arid months, the lands away from the sea are hotter than the coastal tracts, causing a dry land breeze to blow towards the sea during the day which is then longer than the night, thus accelerating the evaporation of brine in the salterns. The average temperatures prevailing in the coastal part of West Bengal and Orissa are shown in Table IV.

TABLE IV — AVERAGE TEMPERATURES (°F.)  
(1940-44)

	SAGAR ISLAND			BALASORE (ORISSA)		
	Max.	Min.	Mean	Max.	Min.	Mean
Dec.	85	56	70	80	56	68
Jan.	81	53	67	81	56	69
Feb.	85	56	71	85	62	73
Mar.	91	66	78	93	69	81
Apr.	92	69	81	97	76	87
May	95	71	83	97	79	88
June	96	75	85	93	79	86
Oct.	89	72		88	73	
Nov.	87	63		84	63	

As is evident from the figures, the coast-land of Bengal in the western part has a mean temperature of about 81°F. (normal diurnal 85°F.) during the months salt is manufactured, and the average mean temperature in Orissa coast is about 79°F. (normal diurnal about 85°F.). Such temperatures with a humidity of 70 per cent are favourable for raising salt from brine.

The maximum temperature of Bombay during January to May varies from 84°F. to 91°F. and that of Madras from 83°F. to 97°F. From the table above it is seen that the maximum temperature of Sagar in West Bengal and Balasore in northern coast of Orissa varies from 80°F. to 97°F.

(v) *Wind Velocities* — Both velocity and dryness of the winds during the salt season affect the evaporation of brine.

It may be stated here that when the wind is dry, and is blowing from the land, its velocity is not relatively important. Likewise, provided the breeze from the sea or marsh land is moderate, however moist it be, it does not materially retard the progress of brine evaporation, especially during the day time when the temperature is high and the air has a high saturation capacity. If the humidity is below 75 per cent during the forenoon, the air can still take up moisture. Sea breeze disturbs the surface of brine in the pans and accelerates evaporation.

TABLE V — AVERAGE WIND VELOCITIES IN MILES PER HOUR (1940-45)

	BOMBAY	BALASORE	SAGAR ISLAND (West Bengal)	MADRAS
Jan.	5.4	1.8	6.6	9.5
Feb.	5.7	2.6	9.0	7.0
Mar.	6.2	3.9	13.5	8.2
Apr.	6.3	5.4	15.7	8.9
May	5.6	5.7	19.7	10.2
June	6.8	4.8	15.9	11.0
Nov.	...	1.6	6.4	...
Dec.	4.5	1.6	7.0	...

The average wind velocities at Sagar and Balasore differ from those of Bombay and Madras (TABLE V). Bombay has a more or less steady wind velocity during the salt season, which is lower than that of Madras. In Sagar Island the wind velocity is higher than that of Madras. The wind velocity in Balasore is, however, poor. Throughout it is very much less than that in Bombay.

### Conclusion

From an examination of the climatic factor and the salinity of brine obtainable along the sea coast of West Bengal and Orissa it may be concluded that the littoral is not unsuitable to salt manufacture. This conclusion is supported by the fact that a flourishing salt industry had been established in these two provinces before the days of East India Company. The question arises whether climate and salinity factors are favourable for salt culture by solar evaporation. The span of the salt season is almost equal to those of Bombay or northern Madras. The only factor for consideration is the rain and storm due to Norwesters. Naupada is equally subject to rain and storm during April and May. It would appear that salt can be raised in West Bengal and Orissa as in Naupada; if it cannot be done entirely by solar evaporation, it should be possible to manufacture salt by combining solar evaporation (70 per cent) with boiling by heating (30 per cent).

The old salt industry in these regions was dependent both on solar and artificial heat, and the process in vogue was different from that now used in southern Orissa and Madras. The people of the coastal regions used salt earth instead of salt water to get concentrated brine, which they boiled to recover salt. This method of leaching the salt earth and boiling the leachate is current on a cottage scale, having been revived after the lifting of the restriction on salt making in 1930. The people in the southern coastal area of Orissa have adopted the Madras practice of raising salt from brine by solar evaporation.

For the commercial manufacture of salt along the coast-land of West Bengal and Orissa, the method as followed in other places of India is recommended. Successful results have been achieved in a few of the West Bengal salt factories which have introduced this method. It is, however, advisable to provide for boiling if the level of production has to be raised.

The sea-board of Contai in the district of Midnapore has been surveyed by the author on behalf of the Provincial Government. It affords vast tracts of waste land, flooded by tidal flows of the sea, highly suitable for salt manufacture. From the land records of the Government, these tracts cover 9,000 acres of land and creeks of which only 150 acres have been developed to salterns by private limited companies. The sea water can be impounded during high tides and utilized, but irrigation pumps will be necessary towards the end of the manufacturing season.

If this area is fully developed, more than 40 lakh md. of salt, the present annual requirement of West Bengal, can be produced. This level of production can be maintained, if provision is made as stand by for boiling during adverse weather conditions.

In the combined process of solar evaporation and boiling, as practised in Burma; the cost of production is a little higher than that in the solar process, but the quality of salt is better and the rate of production can be maintained. The cost of salt production in Contai factories is estimated to be little higher, but as the markets are near, there will be considerable saving on transport.

As to the availability of fuel (coal) for boiling, Calcutta will be the source of supply, and the vessel which would tranship salt from Contai to the port of Calcutta will carry coal on their return voyage. The quantity of steam coal required is about 25 sr. per md. of salt, and the transport of coal to the Contai salt sources will not be uneconomical.

Two firms have been established at Contai for manufacturing salt (combined annual output of 30,000 md.) by solar evaporation. The bigger of these two firms has furnaces to boil saturated saline when necessary. The following data, collected from the factories, will prove useful for developing the areas surveyed by the author.

Average densities of brine on the Contai sea-board are as follows :

TABLE VI—AVERAGE BRINE DENSITY ON  
CONTAI SEA-BOARD

	°Be		°Be
Nov.	1.0	Mar.	2.6
Dec.	1.5	Apr.	2.7
Jan.	1.8	May	2.8
Feb.	2.5	June ( up to 15th )	2.7

TABLE VII—APPROXIMATE RATE OF  
EVAPORATION PER DIEM

	ISCM	
Pond I	2.5-4	0.20
" II	4.8	0.18
" III	8.12	0.17
" IV	12.18	0.16
" V	18.23	0.15

Besides the lands already surveyed on the Contai sea-board, there are suitable lands in the 24 Parganas (Sunderbans). The estuaries to the west of the rivers Hugli, Saptamukhi, Thakuran and Matla are salty during the dry season, and on both sides of each of these estuaries there are lands suitable for raising of salt. In the Sunderbans area about 5,000 acres of waste land will be available, from which 15 to 20 lakh md. of salt can be anticipated. The transport by water will be easy and the prospects for raising salt in this region are very bright.

# German Dyestuff Industry

K. VENKATARAMAN

(Continued from April issue)

## APPENDIX 10

### BLAUTHIOSAURE

*Principal Use* — For DB-Naphthen.

*Equipment* — Homogeneous leaded stirring vessel, 4.5 cu. m. with a cap and descending leaden cooler (reducing vessel), and iron stirring vessel, 6 cu. m. (distilling receiver). Iron stirring vessel, content 7 cu. m., with a coil (condensation vessel). Homogeneous leaded and brick-lined stirring vessel with a coil, content 7 cu. m.; 1 iron press, size 600×900 mm. and 2 wooden presses, size 800×800 mm.

*Process: Reduction to Mercaptan* — Into the reducing vessel are introduced 600 l. of water, 230 kg. of zinc dust and then 225 kg. of "Blausulphochlorid". The temperature is maintained at 20°C. so that no reaction sets in yet. While still cooling, 1,000 kg. of sulphuric acid (73 per cent) are run in. Only when the last third of sulphuric acid runs in, the cooling is stopped, so that the reduction begins. Now, the temperature is raised to 95°C. by heating the vessel. For 3 hr. this temperature is kept. Then cooled to 50°C. and 130 kg. of iron powder introduced; the mercaptan is distilled off by direct steam. Time required: about 15 hr.

*Condensation* — The mercaptan and the water from distillation are pressed out of the receiver into the condensation vessel. Here it is dissolved with 380 kg. of caustic soda solution (33 per cent) and while adding about 300 kg. of ice, it is condensed at 0°-5°C. with 125 kg. of chloroacetic acid. Then it is heated to 95°C. and after adding 10 kg. of clearing carbon it is filtered into the brick-lined stirring vessel by passing through the iron press. The residue contained in the press is then washed out with about 1 cu. m. of water at 70°C. from the condensation vessel. Cooled down to 15°C. and 440 kg. of hydrochloric acid added whereby the "Blauthiosaure" is precipitated. After stirring for 10 hr. the acid is filtered

on the 2 wooden presses, washed with about 1 cu. m. of water and dried at 80°C.

*Process Control Tests* — (1) After finishing the distillation, a sample from the reducing vessel is again distilled with steam, and the distillate mixed with lead acetate. No yellow colouration is allowed (yellow lead salt of mercaptan).

(2) After finishing the condensation, a spot test, on lead acetate paper, shows the presence of non-condensed mercaptan by yellow colouration.

(3) Determination of dry content (99.8 per cent), determination of the melting point and ash.

*Raw Materials, Intermediates & Finished Product Specifications* — The "Blausulphochlorid" has to be further worked up immediately after manufacturing. The finished product has to be clearly soluble in caustic soda solution; m.p., 120°-130°C. White crystals.

## APPENDIX 19

### p'-METHOXY-PHENYL-PARAMINIC ACID

*Principal Use* — For the manufacture of Blue-base F.

*Equipment* — (1) 1 *montejus* for pasting up "p-nitrochloresäure", content 7.5 cu. m.; (2) 1 brick-lined condensation vessel with direct lead of high pressure steam, about 6 cu. m. useful content and connected with a small separator with Raschig rings and water spraying; (3) 1 brick-lined condensation vessel, about 9 cu. m. useful content with direct lead of high pressure steam; (4) 1 brick-lined reducer, 10-11 cu. m. useful content, with direct steam connection; (5) 1 brick-lined precipitating vessel for p'-methoxy-phenyl-paraminic acid, 12 cu. m., and a pump for conveying to the suction filter; (6) 1 brick-lined receiving *montejus* for the condensation mixture, about 8 cu. m.; (7) 2 rubber-lined suction filter receivers for the precipitation of the final liquors, 9 cu. m. each; (8) 1 brick-lined suction filter for the

\*This reference was inadvertently omitted from page 130 of the April issue.



finished product; (9) 1 rinsable "Monstre" press (for iron sludge), 32 frames,  $1,200 \times 1,200$  mm.; (10) 1 "Monstre" press for the precipitate from the mother liquor, 35 frames,  $1,200 \times 1,200$  mm.

**Process** — 772 kg. of "*p*-nitrochlorsaur" (100 per cent, mol. 327.5), as moist neutral product of 40-50 per cent, are pasted up in a *montejus* with water to a paste of 3.3 cu. m. and 85 kg. of magnesium oxide are added while stirring (84 per cent = about 71 kg. of MgO, 100 per cent); this paste is pressed into the brick-lined condensation vessel. To this are added from a pressure vessel 400 kg. of *p*-anisidine in molten condition. The mixture is heated to 160°C. by high pressure steam at 10 atm. pressure and kept at a temperature of 160°-165°C. for 3 hr. The pressure in the condensation vessel is 6 atm. Then the pressure is let off to a separator, filled with Raschig rings and sprayed with water, where parts of non-condensed *p*-anisidine are collected. When the temperature has fallen to 100°-105°C., the contents are pressed off to a brick-lined stirring *montejus*; the main quantity of the magnesium salt of 4'-methoxy-4-nitrodiphenylamine-2-sulphonic acid is already deposited in the hot liquor. The surplus magnesia is neutralized by adding about 100 kg. of sulphuric acid (30°Bé.). The hot condensation mixture is now, in portions, pressed into the brick-lined reducer containing 450-480 kg. of sifted iron filings, with water and a small quantity of sulphuric acid, 30°Bé. The reduction mixture is constantly kept boiling. The reduction of the total nitro-acid is finished when the drop test on filter paper shows no more a yellow, but a purple border. Further, the end of the reduction is tested with Mohr's salt. The reduction finished, the mixture is made slightly alkaline by addition of 85 kg. of soda and filtered boiling hot from the iron sludge through a rinsable "Monstre" press. The iron sludge is washed out with boiling water. The reduction liquid, combined with the washing water, 8.5-9 cu. m., containing about 11 volumes per cent of material, is conducted to a brick-lined precipitating vessel, 2 kg. of zinc dust and 2 kg. of hydrosulphite are stirred in. After the addition of 1,300-1,500 kg. of salt, the liquid is cooled to 20°C. The sodium salt of *p*'-methoxy-phenyl-paraminic acid deposits in well-formed crystals. A sample of the mother liquor shows a sp. gr. of 22°-22.5°Bé. The product is filtered on a suction filter and sucked dry. From the mother liquor, the

free *p*'-methoxy-phenyl-paraminic acid is precipitated in the rubber-lined suction filter receivers by addition of 80-90 kg. of sulphuric acid at 60°Bé. and pressed off in the "Monstre" press. This acid is added to the next reduction. The sodium salt of *p*'-methoxy-phenyl-paraminic acid is, as a rule, 64-68 per cent; mol. wt., 294, and contains 4.5 per cent of sodium chloride. It contains 1 mol. of water of crystallization.

**Yield** — 870-880 kg. of *p*'-methoxy-phenyl-paraminic acid (100 per cent, mol. wt., 294; 91-92 per cent of the theoretical).

**Process Control Tests** — Test for the end of the reduction by the colour of the drop border and with Mohr's salt. Determination of the sp. gr. of the mother liquor after the precipitation of 4'-methoxy-phenyl-paraminic acid. Determination of the percentage of *p*'-methoxy-phenyl-paraminic acid by titration with normal nitrite solution.

**Raw Materials, Intermediates & Finished Product Specifications** — *p*-Anisidine: melting point, 57°C.; magnesia should be 83-85 per cent and as far as possible free from carbonate; "*p*-nitrochlorsaur" must be neutral. Iron filings should be free from coarse elements. *p*'-Methoxy-phenyl-paraminic acid is a grey-violet, finely crystalline product, containing 1 mol. of water of crystallization. Average composition: 65 per cent of *p*'-methoxy-phenyl-paraminic acid, mol. wt. 294 or 69.9 per cent of the sodium salt, mol. wt. 396, 24-25 per cent of water (including water of crystallization) and 4.5 per cent of sodium chloride.

## APPENDIX 22

### "AMIDOL" ACID

**Principal Use** — For producing dyestuffs.

**Equipment** — (1) 1 dissolving vessel for  $\beta$ -naphthol, 4.5 cu. m.; (2) 2 vats for brine, nitrous acid treatment with cooling, 12 cu. m.; (3) 2 leaded transport vessels, 7.5 cu. m.; (4) 3 acid-proof brick-lined rearrangement vessels, 15 cu. m. useful capacity, with twirling stirrers and well working hood; (5) 1 acid-proof brick-lined suction filter with a corresponding rubber-lined receiver; (6) stock and measuring vessel for nitrite solution (40 per cent), stock and measuring vessels for sulphuric acid (30 per cent and 60 per cent), stock and measuring vessel for bisulphite solution (38-40 per cent), stock vessel and vat for weighing caustic soda solution (33.5 per cent); and (7) various receivers for sulphureous acid absorption.

*Process* — In a *montejus* 576 kg. of  $\beta$ -naphthol scales are dissolved at about 33°C. in 3,000 l. of water and 478 kg. of caustic soda solution. The solution is made up to 4,500 l. and pressed into the vat for treatment with nitrous acid. Here the  $\beta$ -naphthol solution is cooled with brine to 3°C. The  $\beta$ -naphthol is precipitated in a finely divided form by adding about 560 kg. of iron-free sulphuric acid (30 per cent strength) through a distributing nozzle. The liquid must have a slightly alkaline reaction to phenolphthalein. In order to obtain, as far as possible, a fine and homogeneous distribution of the  $\beta$ -naphthol, the mixture is stirred for some time, and then 690 kg. of nitrite solution of 40 per cent strength are added. Now, at 5°-8°C., further 680 kg. of iron-free sulphuric acid of 30 per cent strength are slowly run in through the distributing nozzle. The liquid must now have a distinctly acid reaction to Congo paper and must indicate nitrite. The nitroso- $\beta$ -naphthol precipitates as a slightly yellow, finely divided product. After additional stirring at not more than 8°C. for several hours, the excess acid is neutralized by adding 30-40 kg. of caustic soda solution. The liquid must have a neutral reaction. 2,400 kg. of sodium bisulphite solution are run into this paste of nitroso- $\beta$ -naphthol. The temperature rises to 18°-20°C. and a solution is obtained showing a few dark, flocky, separated particles. After adding 500 kg. of rock salt, the solution is pressed into the rearrangement vessel containing 5 cu. m. of the mother liquor from the preceding batch with a content of 5.5-6 per cent by volume of sulphuric acid. The mixture (about 13 cu. m.) is acidified with 700 kg. of sulphuric acid of 60 per cent strength and heated to 38°C. by direct steam. The temperature slowly rises to 50°-52°C. The "Amidol" acid begins to separate as a slightly grey crystalline precipitate. The precipitation is completed by allowing to stand for about 36-40 hr. and by occasional stirring to avoid complete solidification of the mass. Then the mass is thoroughly stirred and the "Amidol" acid is discharged on an acid-proof suction filter and sucked off. The product is washed free from acid with about 4-5 cu. m. of water and the neutral product of about 25 per cent strength, mol. wt., 239, is worked to "Diazoamidol" acid. For dispatch, the product is filtered by suction as dry as possible (32-36 per cent).

*Yield*—765-770 kg. of "Amidol" acid of 100 per cent strength, 80-81 per cent of the theory.

*Process Control Tests* — Test as to complete dissolution of  $\beta$ -naphthol in caustic soda solution. Test of the nitrous acid treatment as to an acid reaction of the liquid and as to an excess of nitrite solution. Test of the "Amidol" acid as to its neutral reaction. Determination of the content of the moist "Amidol" acid by diazotizing in neutral solution with copper sulphate as catalyst.

*Raw Materials, Intermediates & Finished Product Specifications* —  $\beta$ -naphthol (scales) must be soluble, as clear as possible, in caustic soda solution; m.p. 120°C., tech. product of 100 per cent strength.

Nitrite solution of 40 per cent strength.

Bisulphite solution of 38-40 per cent strength.

Sulphuric acid of 30 per cent strength; must be as free from iron as possible.

Caustic soda solution of 33.5 per cent strength, commercial product.

Sulphuric acid of 60 per cent strength, commercial product.

Rock salt, commercial product.

The moist "Amidol" acid is a slightly grey, crystalline product in paste form of about 25 per cent strength.

The product, well sucked off for dispatch, is 32-36 per cent.

## APPENDIX 23

### DIAZOAMIDOL-ACID

*Principal Use* — For dyestuffs.

*Equipment* — (1) 2 pasting vessels for the amidol-acid, 7.5 cu. m., one of them rubber-lined; (2) 1 diazotizing vat with high-speed stirrer and brine cooling; (3) 2 leaded receivers for the diazo solution, 7.5 cu. m.; (4) 2 small washable clarifying presses, each containing 18 chambers, 80 × 80 cm.; (5) 2 acid-proof, brick-lined precipitating vessels with swirl-stirrer, 10.5 cu. m.; (6) 2 homogeneously leaded pendulum centrifuges; (7) 1 acid-proof suction filter with corresponding receiver.

*Process* — 700 kg. of amidol acid (100 per cent; mol. wt., 239) as a moist neutral paste of about 25 per cent are stirred with water so as to obtain about 3,000 l. of a 21-21.5 per cent paste. The diazotizing vessel, fitted with high-speed stirrer, contains 1,000-1,200 l. of water of the washing liquor (which is obtained by washing out the residue of the preceding operation), 2.5 kg. of crystallized copper sulphate dissolved in some litres of water and 540 kg. of nitrite solution

(40 per cent) respectively. To this the 3,000 l. of amidol acid paste are pressed in the course of about 3 hr. Within this time the slowly rising temperature must not exceed 25°C. The amidol acid paste totally dissolves — while forming the diazo salt — to a dark, golden-yellow solution. Care is taken that there always exists an excess of nitrite. Then the solution is rendered slightly acid to litmus by adding sulphuric acid (30 per cent) rendering the dark flocculent residue easily filterable. The solution is filtered through a clarifying press and the residue washed out with warm water at 40°-45°C. The first portion of the washing water (about 500 l.) is combined with the clear filtrate. The bulk (about 1,000 l.) is used again in the following diazotization: the filtrate combined with the first washing water is acidified by slowly adding 1,350 kg. of sulphuric acid (30 per cent) within about 8 hr. at 25°-28°C. The diazotized amidol acid separates as well-crystallized, greyish-orange product. Separation is finished as soon as the mother liquor indicates 5.5-6 per cent by volume of sulphuric acid. The separated diazoamidol acid is centrifuged from the mother liquor on the homogeneously leaded pendulum centrifuges and obtained as a sandy, greyish-orange product with a content of 86-88 per cent (mol. wt., 250). For avoiding losses the mother liquor passes from the centrifuge to a suction filter. For working up to chlorodiazoamidol acid the product must be dried below 50°C. when a brownish dusting product (92-93 per cent; mol. wt., 250) is obtained.

**Yield** — 688-695 kg. of moist diazoamidol acid calculated as a product of 100 per cent (94-95 per cent of the theory).

#### *Process Control Tests* —

Test of the amidol acid paste as to neutrality.

Test of the diazotizing process as to presence of nitrite solution.

Test as to the total separation of the diazoamidol acid by determination of the acidity of the mother liquor.

Testing the content of the moist and of the dry finished product by determination of the diazo nitrogen evolved on decomposition with cuprous chloride solution.

#### *Raw Materials, Intermediates & Finished Product Specifications* —

Nitrite solution of 40 per cent strength.

Sulphuric acid of 30 per cent strength.

Crystallized copper sulphate: commercial quality.

Acidity of the mother liquor after separation: 5.5-5.6 per cent by vol.

The product is clearly soluble in caustic soda solution.

The diazoamidol acid contains 1 mol. of water of crystallization.

## APPENDIX 25

### NITRO-ETHYLIMIDE BASE

**Principal Use** — After reduction to aminoethylimide base for Sirius Light Blue FFRL.

**Equipment** — (1) boiler, 1 cu. m., cast iron vessel with jacket of silica stones, stirrer and lid coated with V<sub>2</sub>A steel; (2) 1 pressure suction filter, 1.5 sq. m., brick-lined, with receiver; and (3) 1 stone suction filter with nozzle, 2.5 sq. m.

**Process** — Introduce 200 kg. of ethylimide base (N-ethyl carbazole) at ordinary temperature into 180 kg. of chlorobenzene and dissolve by stirring for 1 hr. Temperature 20°-25°C. 305 kg. of nitric acid (35.5 per cent) are run in within about 5 hr. Temperature 27°-28°C. (cooled by water or brine). After stir overnight at a temperature between 25°-30°C. (a sample should have a melting point of 127°-130°C.) (Test a). Cool to 10°C. and stir at 100°C.; after 8-10 hr. filter on the pressure suction filter and wash thrice, each time with 10 kg. of chlorobenzene. Then bring the batch on the stone suction filter and wash there 2-3 times, each time with 250 kg. of Leonil SB-solution of 1 per cent strength (Test b). Finally, wash until neutral with 50 kg. of water. Dry in the circulating air chamber at a temperature of 50°-60°C.

**Process Control Tests: Test a** — Filter the sample by suction, wash it for a short while with chlorobenzene and then wash thrice until neutral with Leonil water of 2 per cent strength and finally with water. Dry on clay tile, 129°-130°C.

**Test b** — Distil 100 gm. of the material on the filter with steam in order to test whether the chlorobenzene has been completely washed out with Leonil and water.

**Raw Materials, Intermediates & Finished Product Specifications** — Ethylimide base (N-ethyl carbazole): point of solidification, 64.5°-65°C., clearly soluble in chlorobenzene. The nitro-ethylimide base is beautiful yellow crystals, m.p., 129°-129°C. The product must be completely neutral to Congo paper.

## APPENDIX 26

## CARBAZANIL

*Principal Use* — Starting material for carboxazine, for Sirius Supra Blue F3RL.

*Equipment* — (1) Brick-lined stirring vessel, 800 l., with steam jacket and leaded reflux condenser; and (2) filter press, 800 × 800 mm.

*Process* — 8.5 kg. of chloranil are introduced into a suspension of 125 kg. of spirit of 50 per cent by volume, 12 kg. of amino-carbazole and 6 kg. of anhydrous sodium acetate within  $\frac{1}{2}$  hr. at room temperature. The mixture is heated to boiling and kept at boiling temperature for 6 hr. while stirring and refluxing. After cooling down to 25°-30°C., the product is filtered off and washed with 25 kg. of spirit of 25 per cent by volume. Then it is freed from ash by washing at first for 2 hr. with hot water and finally for  $\frac{1}{2}$  hr. with cold water. The pressed cake is blown with nitrogen until dry and dried in the vacuum-drying closet at 65°-70°C. The spirit solutions are regenerated.

## APPENDIX 27

## "TETRA ACID"

*Principal Use* — Starting product for producing Indanthren Printing Brown B and 5R and Indanthren Scarlet GG.

*Equipment* — Brick-lined (2 layers) iron vessel, 16 cu. m. with wooden stirrer and leaded cover; open, brick-lined suction filter with stone filter, 4 sq. m.; cemented iron vessel, 5 cu. m., with stirrer; clarifying filter; iron vessel, 6 cu. m. (clarifying receiver); 2 iron filter presses, 800 × 800 mm. each; rubber-lined iron vessel, 16 cu. m., with stirrer.

*Process* — In the brick-lined vessel containing a moist paste of "diketimide" (250 kg. of dry product), is added a mixture of 2 cu. m. of water and 2,217 kg. of concentrated hydrochloric acid. The mixture is heated to 40°C. by blowing in steam, and 145 kg. of sodium chlorate are carried in within 2 hr. The temperature rises to 60°C. After stirring for 8 hr., the mixture is made up to 10 cu. m. by addition of cold water; the product is sucked off on the suction filter and washed to neutral reaction.

In the cemented iron vessel with stirrer, 2,130 l. of water, 385 kg. of caustic soda solution (22 per cent) and 650 kg. of sodium hypochlorite liquor are heated to 40°C. and one-third of the suction filter material is carried in. The temperature rises to 50°C.

and is maintained for 2 hr. The mixture is neutralized by addition of 50 kg. of bisulphite solution, clarified through a clarifying filter into the iron vessel (6 cu. m.) and washed with 4 cu. m. of hot water. This water is again used for the following batch.

The clarified liquor is carried back into the oxidation vessel, 60 kg. of caustic soda solution (22 per cent) and 575 kg. of sodium hypochlorite are added and the mixture is oxidized at 30°-40°C. by introducing about 120 kg. of potassium permanganate, added shovel by shovel. The temperature rises to 70°C. After 20 min., the excess of potassium permanganate is destroyed by addition of 50 kg. of bisulphite and the manganese mud is filtered in 2 filter presses. The filtrate runs into the rubber-lined vessel. The manganese mud is washed hot, pasted again, filtered and washed until tetra acid can no longer be detected in the filtrate. In the filtrate the tetra acid is precipitated by addition of 1,000 kg. of crude hydrochloric acid, sucked off on a suction filter and washed with 800 l. of water and 20 l. of hydrochloric acid (1 per cent). The tetra acid is dried at 80°-90°C.

*Process Control Tests*— (a) After the reaction with sodium chlorate, a sample is sucked off, washed first with cold water to neutral reaction and then with hot water. In this filtrate no "diketimide" must be precipitated by addition of salt. The fluorescence effected by exposing to the rays of a quartz-lamp must only be weak-bluish, compared with the test substance. When "diketimide" is present, 10-20 kg. of sodium chlorate and 300-500 kg. of hydrochloric acid are added.

(b) After the oxidation by potassium permanganate, a sample spread on filter paper must have a green bleeding which should not disappear after stirring for 20 min. In an acidified sample of the filtrate the tetra acid has to precipitate in well-formed crystals.

(c) After the precipitation of the tetra acid, the filtrate must still turn Congo paper blue.

(d) When further acid is added to a sample of the filtrate, no tetra acid must be precipitated.

(e) When a sample of the manganese mud is extracted with hot dilute caustic soda solution, the acidified extract must not show any precipitation.

*Raw Materials, Intermediates & Finished Product Specifications* — The finished product is condensed with orthamine and glacial acetic acid to Indanthren Scarlet GG and

the latter is tested as to its dyeing properties. The yield should amount to 136 per cent of the used tetra acid. Tetra acid should be tested for organically bound chlorine, iron and ash content.

## APPENDIX 29

### PYRENIT S

*Principal Uses* — Starting material for Pyram, intermediate for Siriuslichtblau F3GL.

*Equipment* — Clay pot with V<sub>2</sub>A steel high-speed stirrer, 700 l.; brick-lined suction filter; brick-lined receiver with connection to the vacuum piping; and swinging mill.

*Process* — In the clay pot, 100 kg. of pyrene (ground on the Colloplex mill and afterwards on the swinging mill) is stirred 405 kg. of nitric acid of 22 per cent strength for 8 hr. at 40°-45°C. (test), then sucked off, washed until neutral and dried. The mother liquor is stored and used again.

*Process Control Test* — The m.p. must be 148°-150°C.

*Raw Materials, Intermediates & Finished Product Specifications* —

Pyrene, free from iron.

Finished product: yellow-brown powder, m.p., 148°-150°C.

## APPENDIX 30

### PYREN-CHLORID

*Principal Use* — Starting material for pyrenequinone.

*Equipment* — Enamelled cast-iron stirring vessel, 2 cu. m., in a diphenyl bath, with coil for steam of 15 atm. (reaction vessel); brick-lined iron suction filter, 2.5 sq. m., with brick-lined receiver; brick-lined iron stirring vessel, 4.5 cu. m., with iron cooler and iron separator, 6 cu. m.; brick-lined iron suction filter, 3 sq. m.

*Process* — In the reaction vessel, 1,350 kg. of trichlorbenzene are heated to 180°C. While stirring, one-third the tetrachlorpyrene tetrachloride (corresponding to 180 kg.) and 36 gm. of ferric chloride are introduced, at first in small portions, until splitting off of hydrochloric acid occurs. The reaction finished, the product is sucked off on the suction filter at 30°C. and washed with 400 kg. of trichlorbenzene. The mother liquor is used for the 2 following batches. The material from the suction filter is freed from the solvent in the distiller, sucked off on the second suction filter, washed until neutral

and dried at 60°-70°C. The remaining two-thirds of the tetrachloride is treated in the same way. The yield of 3 batches amounts to 400 kg. The trichlorbenzene is recovered in a special equipment. The loss of solvent amounts to 20 per cent.

*Process Control Tests* — Final point of the splitting off; termination of the evolution of hydrogen chloride.

*Raw Materials, Intermediates & Finished Product Specifications* — Hexachlor-pyrene is a yellow, crystalline product, m.p., 360°-375°C. The pure product melts at 382°C.

## APPENDIX 31

### PERCHLORPYRENE

*Principal Use* — Starting material for hexachlor-pyrene.

*Equipment* — Brick-lined, homogeneously leaded stirring vessel, 3 cu. m., with leaded heating candle (reaction vessel); brick-lined iron suction filter, 2.5 sq. m., with brick-lined receiver; enamelled, cast-iron stirring vessel, 2 cu. m., in a diphenyl bath with coil for steam of 15 atm. (pasting up vessel).

*Process* — Into the reaction vessel containing 1,800 kg. of trichlorbenzene, 120 kg. of pyrene and 120 gm. of iodine, 500 kg. of chlorine are passed in at 108°-110°C. within 24 hr. After cooling to 90°C., a further quantity of 120 kg. of pyrene and 120 gm. of iodine are introduced and the chlorination is continued at 108°-110°C. (12-15 kg. of chlorine per hour). In all, 480 kg. of pyrene are chlorinated, the chlorine stream finally amounting to 8 kg. per hour. The hydrogen chloride gases together with the excess of chlorine go through separators to the chimney. Duration of the chlorination: 6 days. The product is sucked off at 30°C. on the suction filter and washed with 400 kg. of trichlorbenzene from the reaction vessel. The material from the suction filter is stirred in the pasting-up vessel with 63 kg. of trichlorbenzene, sucked off on the same suction filter and washed with 200 kg. of trichlorbenzene and processed to hexachlor pyrene.

The filtrates and washing liquors of trichlorbenzene are worked up in a special equipment. Yield: 540 kg.

*Process Control Tests* — Final point of the chlorination: 50 c.c. of the suspension are filtered and the white residue on the filter is washed with carbon tetrachloride; the product shall be completely soluble in 10 times its quantity of chlorobenzene. Tetrachlorpyrene is insoluble. In 10 times its

quantity of nitric acid (sp. gr. 1.4), no change of colouration should occur. Traces of tetrachlorpyrene give a brown colouration with nitric acid.

## APPENDIX 32

### "PYRANIL", CRUDE

*Principal Uses* — Starting material for "Leucopyranil"; intermediate product for Sirius Light Blue F3GL.

*Equipment* — A 3 cu. m. enamelled boiler with steam jacket, stirrer; spirit mother liquor vessel; spirit regenerating apparatus; and a "Monstre" press, 1,200 × 1,200 mm.

*Process* — In the 3 cu. m. reaction vessel, 145 kg. of "Pyram", 102 kg. of chlorophenol, 104 kg. of chloranil and 116 kg. of anhydrous sodium acetate are condensed in 1,200 kg. of spirit (95 per cent) at first at 0°C. (cooling with 200 kg. of solid carbonic acid), then at 20°-25°C. for 12 hr. Then filtered in the "Monstre" press. Finally, the filtered product is washed with water until neutral and free from spirit. The moist product is dried at 90°-100°C. in a vacuum drying closet. The spirit mother liquor and the washing waters are collected and distilled.

*Raw Materials, Intermediates & Finished Product Specifications* —

- (1) Aminopyrene ("Pyram"): m.p., 114°-116°C.
- (2) Chloranil: not below 99 per cent.
- (3) *o*-Chlorophenol: about 90-97 per cent of *o*-chlorophenol.
- (4) Sodium acetate, anhydrous.
- (5) Spirit not below 90 per cent by weight.

## APPENDIX 33

### "PYRANIL" PURE

*Principal Uses* — Starting material for "Pyroxazine"; intermediate product for Sirius Light Blue F3GL.

*Equipment* — A 2 cu. m. enamelled vessel with oil circulation heating; 7.5 cu. m. expeller, cooler; recovering equipment for nitrobenzene; and a suction filter.

*Process* — In the enamelled vessel, containing 1,390 kg. of nitrobenzene, 110 kg. of "Leucopyranil" are quickly introduced at 190°C. and after-stirred for 45 min. Then the reaction mixture is pressed into the expeller with 3,000 l. of water and the nitrobenzene is driven off by steam. This product is then sucked off on a suction filter and washed free from ashes by means of hot water.

*Process Control Tests* — After the reaction the microscopic structure shall show fine, hair-shaped, brown crystals and no longer colourless crystals of "Leucopyranil".

*Final Test* — Dry weight and ash determination. Reduction to the "Leucopyranil" according to prescription. The yield in per cent of the amount used gives the degree of purity for "Pyranil" pure.

*Raw Materials, Intermediates & Finished Product Specifications* — "Leucopyranil" must be absolutely dry.

Finished product: fine, hair-shaped, crystallized, brown product, m.p., 331°-332°C.

## APPENDIX 34

### PYRAM

*Principal Use* — Intermediate product for pyranil (intermediate for Sirius Light Blue F3GL).

*Equipment* — Iron stirring vessel with jacket, 4 cu. m., with reflux condenser, iron pressure filter with removable lid and cloth filter, 3 sq. m.

*Process* — In the 4.5 cu. m. iron stirring vessel 320 kg. of Pyrenit S are dissolved in 820 kg. of spirit (at least 94 per cent) by refluxing for 2 hr. 260 kg. of hydrosulphide (with 96 kg. of H<sub>2</sub>S) are dissolved in 130 l. of water in casks; the solution is siphoned off from the contaminations and runs into the Pyrenit solution within 3 hr. By the violent reaction the batch is kept boiling. After running in is finished, refluxing is continued for further 3 hr., then 600 l. of water are filled in and the whole is cooled to 20°C. Pyram crystallizes within 15 hr., is filtered off on the pressure filter, washed with 240 kg. of spirit (50 per cent) and afterwards with about 1,000 l. of water until neutral, and dried at 60°-70°C.

*Raw Materials, Intermediates & Finished Product Specifications* — Pyrenit: m.p., 144°-145°C. (pure product 147°C.).

Finished product: m.p., 116°-117°C.

## APPENDIX 35

### "LEUCOPYRANIL"

*Principal Uses* — Starting material for "Pyranil pure"; intermediate product for Sirius Light Blue F3GL.

*Equipment* — A 2 cu. m. enamelled vessel, stirrer, circulation cooler; pressure filter, leaded and brick-lined; receiver, 4.5 cu. m., leaded; and apparatus for the recovering of chlorobenzene.

**Process** — In the 2 cu. m. enamelled vessel 1,320 kg. of chlorobenzene and 135 kg. of crude "Pyranil" are heated to boiling. Then 30 kg. of phenylhydrazine run in in the course of 1 hr. (test). The reaction mixture is cooled to 70°C., pressed on the suction filter and after-washed twice with 180 kg. of 80°C. hot chlorobenzene. The product is steamed on the suction filter until free from chlorobenzene; after cooling, dried in the drying closet at 70°C. The chlorobenzene mother liquors are worked up in the chlorobenzene recovery apparatus.

**Process Control Tests** — After the reaction a sample taken out of the vessel is filtered on a suction filter at 70°C. in the laboratory. The product shall look light-grey to light-yellow. Microscopic structure: colourless leaflets. In case brown needles of the crude "Pyranil" are still present, further phenylhydrazine must be added to the batch.

**Raw Materials, Intermediates & Finished Product Specifications** —

(1) "Pyranil", crude: test in the laboratory.

(2) Phenylhydrazine, supplied freshly distilled, b.p., 233°C.

(3) Finished product: weakly, yellowish to grey crystalline product.

#### APPENDIX 41

##### INDANTHREN BRILLIANT ORANGE GR & INDANTHREN BORDO RR

**Principal Uses** — For printing and dyeing cotton and artificial fibre, Indanthren Bordo besides as mixed dyestuff.

**Equipment** — Iron stirring vessel with hose, 4.5 cu. m., brick-lined heatable pressure filter with stirrer that may be lifted and lowered, stone filter bottom, 3 sq. m., iron stirring vessel with hose, 2 cu. m., rubber-lined stirring vessel, 4.5 cu. m., horizontal iron vessel, 3 cu. m., iron stirring vessel with jacket, 4 cu. m., with iron condenser and iron receiver, 6 cu. m., brick-lined stirring vessel, 7 cu. m., 2 wooden presses 1,200 × 1,200 mm.

**Process** — In the iron stirring vessel 400 kg. of caustic potash powder are dissolved at 40°C. and 2,000 kg. of spirit and 200 kg. of Indanthren Scarlet GG base P are filled in at 75°C. and are held for 1 hr., then cooled to 20°C. The separated potassium addition product of the Indanthren Brilliant Orange is filtered on the suction pressure filter. In the 2 cu. m. iron stirring vessel the suction

material is added to 420 kg. of spirit (83 per cent) and 200 kg. of caustic potash lye, heated to 75°C., cooled to 20°C. and again filtered on the pressure filter. Washed in 3 portions with a washing spirit consisting of 480 kg. of spirit (95 per cent), 75 kg. of water and 225 kg. of caustic potash lye, produced in the same iron stirring vessel of 2 cu. m. The suction material, Indanthren Brilliant Orange, is hydrolysed in 1-1½ hr. in the rubber-lined 4.5 cu. m. stirring vessel in 3 cu. m. of warm water at 60°C. and the obtained dyestuff is filtered hot into a wooden press and is washed neutral and filled into wooden barrels. The spirit filtrates are collected in the horizontal 3 cu. m. iron vessel and pressed off to the 4 cu. m. iron stirring vessel, in which the spirit is distilled off and collected in the 6 cu. m. iron spirit receiver. The residue, containing the Indanthren Bordo, is slowly pressed off (foaming) into the 7 cu. m. brick-lined stirring vessel in which 1,000 l. of hydrochloric acid crude and 1,000 l. of water of 80°C. are added. The reaction should finally be slightly acid. After stirring for another 1 hr., the dyestuff is filtered to the second wooden press, 1,200 × 1,200 mm., washed neutral and filled into wooden barrels.

**Process Control Tests** — (a) In the second suction material small red points indicate unconverted scarlet. (b) After acidifying the Bordo residue the reaction finally has to be acid to Congo.

**Raw Materials, Intermediates & Finished Product Specifications** —

(a) If difficulties occur, the "tetra acid", already used for Indanthren scarlet, must be examined in a laboratory batch.

(b) The spirit is spindled.

(c) The Indanthren Brilliant Orange GR is examined as double paste in printing, the Indanthren Bordo RR as suprafix paste also in printing compared with standards. The yields are fixed by the weight of the filter cakes and by dry determination. The yield of Brilliant Orange should be 59 per cent. Indanthren Brilliant Orange GR double paste contains 10 per cent pure colour. Indanthren Bordo RR suprafix double paste contains 20 per cent pure colour.

#### APPENDIX 42a

##### INDANTHREN BROWN GR

**Principal Use** — Vat dyestuff for cotton.

**Equipment** — An iron melting vessel of 8 cu. m. provided with stirrer and high



pressure water heating; an iron diluting vessel of 9 cu. m.; and a "Monstre" press.

*Process* — 1,800 kg. of caustic potash are introduced into the melting vessel heated to 140°-150°C. Then the trianthrimide is introduced into the vessel in the course of 4 hr. and heated to 190°-195°C. within 4-5 hr. While maintaining this temperature the whole is stirred for 8 hr. (Sample 1 to be taken). After cooling down to 150°-160°C., water is slowly added until the vessel is filled completely. The contents are allowed to run into the vessel of 9 cu. m. which is then filled up with water. The product is oxidized at 30°-40°C. by introducing air while stirring (Sample 2 to be taken). Then the whole is filtered into the "Monstre" press and washed with water until neutral.

*Process Control Tests: Sample 1* — After the melt is finished, a test portion should be capable of being vatted completely without leaving any residue.

*Sample 2* — Before filtering the melt, a test portion dropped on filter paper must show a bleeding coloured only weakly yellow.

*Final Sample* — An examination is made by producing a dyeing and comparing with the type.

*Raw Materials, Intermediates & Finished Product Specifications* — Final product: brown paste, its vat being reddish brown.

#### APPENDIX 43

##### INDANTHREN TURQUOISE BLUE GK

*Principal Use* — Dyestuff for dyeing cotton.

*Equipment* — 1 enamelled batch vessel, 2 cu. m., with jacket; 1 brick-lined decomposing vessel, 10 cu. m.; 1 filter press, 1,000 × 1,000 mm.

*Process* — In the batch vessel, 750 kg. of chlorosulphonic acid are heated to 35°C. Within 3-4 hr. 127 kg. of "acridone acids" are introduced through a sieve at a temperature of 35°-40°C. The mixture is then stirred for 2½ hr., and when everything is dissolved, the solution is then pressed into the decomposing vessel with 4 cu. m. of water and 2,000 kg. of ice. The temperature must not exceed 25°C. The mixture is stirred for 2 hr., filtered in the filter press and washed until neutral with water at 60°C. Yield: 114 kg. The colour of the solution in concentrated sulphuric acid is brownish yellow. The vat colour is brown;

by transmitted light, dull violet. The powder is of 76.8 per cent strength, the paste of 10 per cent strength.

*Process Control Tests* — After introducing and stirring, a sample is tested as to complete solution by rubbing in the hands.

#### APPENDIX 50

##### INDANTHREN PRINTING BROWN B BASE

*Principal Use* — Printing dyestuff for cotton.

*Equipment* — 1 brick-lined autoclave, 3 cu. m., heatable, with enamelled stirrer; 1 brick-lined, box-shaped suction filter with stoneware filter, 2.5 sq. m. filtering area.

*Process* — In the autoclave 124 kg. of naphthalenetetracarboxylic acid (tetra acid), 170 kg. of ethoxy-1.2-phenylenediamine (ethoxyorthamine) and 80 kg. of 30 per cent acetic acid are heated at 165°C. under 6 atm. excess pressure for 16 hr. while stirring (Test 1). Then the mixture is cooled to 30°C., sucked off, washed with water until neutral and dried in the drying closet at 100°C. For standardizing against commercial article the product dried and ground is first dissolved in sulphuric acid and reprecipitated.

*Process Control Tests: Test 1* — A sucked-off sample from the vessel is extracted with boiling dilute caustic soda solution and filtered. When the filtrate is acidified, flakes of tetra acid must no longer precipitate.

*Raw Materials, Intermediates & Finished Product Specifications* —

Tetra acid }  
Ethoxyorthamine } test for dyestuff.

Finished product: brown powder, dissolves in conc. sulphuric acid to a red solution; vat, wine-red.

#### APPENDIX 51

##### INDANTHREN PRINTING BROWN 3R

*Principal Use* — Dyestuff for printing cotton.

*Equipment* — An iron vessel of 7 cu. m. provided with heating and cooling coil; an enamel vessel of 2 cu. m.; and a "Monstre" press.

*Process* — 100 kg. of Indanthren Printing Brown 3R base well made into a wet paste are introduced into the vessel of 7 cu. m. containing 4,000 kg. of alcohol (20 per cent); 125 kg. of caustic soda solution (35 per cent) are added and the whole is heated



to 70°C. Then 60 kg. of hydrosulphite powder are introduced. The reduction is performed at 80°C. (Sample 1). After cooling down to 25°-30°C., CO<sub>2</sub> is introduced until the leuco compound of the dyestuff is completely precipitated (Sample 2 to be taken). This takes about 4 hr. The whole is brought into the "Monstre" press, washed with cold water until neutral and pressed. The filtrate and the first washing water are collected in order to recover the alcohol.

The leuco compound of the dyestuff is introduced into the vessel of 2 cu. m., mixed with 5 per cent of caustic soda (calculated on dry dyestuff) used in the form of a lye. The whole is heated to 50°C. and air is blown in. The oxidation is finished after about 3 hr. The dyestuff may be adjusted to conform with the Suprafix paste.

*Process Control Tests:* Sample 1 — The vat must be clear apart from some flakes.

Sample 2 — A drop test on filter paper must not produce a halo that turns brown in air. The filtrate when mixed with bisulphite must not show any precipitate.

*Final Test* — A sample of the oxidized dyestuff is sifted through a screen of 250 mesh/sq. cm., then adjusted to a content of 16 per cent of dyestuff and tested by printing. If the print is uneven, oxidation was not sufficient. If the fixation of the dye is insufficient, iron chloride may be added.

#### APPENDIX 61a

##### ANTHRASOL BROWN IRRD

*Principal Use* — For printing and dyeing textiles.

*Equipment* — Iron stirring vessel with jacket, 2 cu. m. (esterification vessel); iron stirring vessel with jacket, 7.5 cu. m. (distiller), with iron condenser and iron receiver, wooden filter press 800 × 800 mm.; iron stirring vessel with jacket, 7.5 cu. m. (salting out vessel); open iron suction filter, 3 sq. m., with cloth filter and iron receiver; and an iron stirring vessel with high-speed stirrer and lower outlet, 0.5 cu. m.

*Process* — Into 750 kg. of pyridine II contained in the esterification vessel 175 kg. of chlorosulphonic acid is run in at 15°-20°C. within 3-4 hr. Then 100 kg. Indanthren Brown RRD (100 per cent), dried and ground, are introduced and after stirring for a short time 60 kg. of electrolytic iron are added. The mixture is heated to an internal temperature of 40°C. by 60°C.

warm water. After heating has been stopped the temperature still mounts to 50°C. and is kept there. After about 3 hr. the esterification is complete.

The batch is pressed by nitrogen into the distiller, which contains a solution of 270 kg. of soda in 3,500 l. of water. After addition of 0.5 kg. of rape seed oil (or other anti-frothing agent) the pyridine is distilled off into an iron receiver at 30°-35°C. under vacuum (duration 10-14 hr.).

After distillation is over, the product is filtered off in the 800 mm. wooden filter press at about 20°C. The filtrate runs into the sewer; it must contain only traces of anthrasol. The crude ester in the same 7.5 cu. m. distiller is pasted up with 5,500 l. of water, some soda and 10 kg. of kieselguhr, heated to 90°C. and filtered into the 7.5 cu. m. iron salting-out vessel through a wooden filter press, pre-heated by steam. The filter product is washed with hot water, until anthrasol can no longer be detected in the filtrate. The wash water at first runs to the mother liquor and then into the distiller, where it is used for the next batch. The filtrate is cooled to about 30°C. while stirring weakly, salted out with 6 per cent of salt and cooled to 20°C. The ester salted out is sucked off on the open iron suction filter (duration 15-20 hr.). The filtrate runs into an iron receiver. The suction filter product is made into a paste with 6 kg. of 33 per cent caustic soda solution in the 0.5 cu. m. iron stirring vessel and dried at 55°-60°C., at first under normal pressure and then in the vacuum. The manufactured article is obtained with a net content of about 55 per cent. The dry material is ground in a cross-beater mill and standardized against type (about 50 per cent of pure dyestuff) with sodium-dimethyl-aniline-sulphonate and sodium sulphate.

In order to dry the pyridine, its aqueous solutions are azeotropically distilled in a special equipment after addition of benzene and then redistilled. The loss amounts to 8-10 per cent.

*Process Control Tests* — (a) After esterification is finished, a sample from the esterification vessel is added to a soda solution, sucked off at 50°-60°C. and washed with warm water. The filter residue is tested for absence of vat dyestuff by vatting.

(b) Before the distillation is started, a test as to alkaline reaction to phenolphthalein must be made.

(c) The distillate is tested for anthrasol with acid and sodium nitrite.

(d) Distillation is finished as soon as the pyridine content of the distillate is <1 per cent.

(e) Before clarifying, a test as to complete dissolution of anthrasol is necessary. If required, water has to be added.

(f) The residue on the paste is tested for anthrasol by extracting with hot water and to vat dyestuff by vatting.

(g) A sample of the anthrasol salted out is sucked off and the filtrate is tested for anthrasol with nitrite and acid.

*Raw Materials, Intermediates & Finished Product Specifications* — Vat dyestuff, pyridine and iron have to be tested in the laboratory by esterifying a sample.

In the finished product the dyestuff content is determined by gravimetric analyses with iron chloride and acid. The yield is indicated as pure dyestuff of 100 per cent.

A sample is standardized against Anthrasol Brown IRRD Powder with 10 per cent of sodium dimethyl-aniline-sulphonate and sodium sulphate and compared with the type by textile printing. Anthrasol Brown IRRD Powder contains 50 per cent of pure dyestuff. For testing the fastness to storing, a sample is maintained at 55°C. for 3 weeks; after this time no decomposition must appear.

#### APPENDIX 61b

##### ANTHRASOL BRILLIANT PINK 13B

*Principal Use* — For printing and dyeing textiles.

*Equipment* — Iron stirring vessel with jacket, 2 cu. m. (esterification vessel); iron stirring vessel with jacket, 7.5 cu. m. (distilling vessel), with iron cooler and iron receiver; wooden filter press 800×800 mm.; iron stirring vessel with jacket, 7.5 cu. m. (salting-out vessel); open iron suction filter, 3 sq. m., with cloth filter and iron receiver; and an iron stirring vessel with high-speed stirrer and lower outlet, 0.5 cu. m. (conche).

*Process* — In the esterification vessel, 180 kg. of chlorosulphonic acid are run into 1,000 kg. of pyridine II within about 3 hr. at 15°-20°C. Then 100 kg. of Indanthren Brilliant Pink 3B of 100 per cent strength, dried and ground, are introduced and, after stirring for a short time, 80 kg. of electrolytic iron are added. The mixture is heated with water of 60°C. to an inner

temperature of 40°C. After the heating is finished, the temperature rises to 48°-50°C. and is kept at that level. After 2-3 hr. the esterification is finished. The batch is pressed with nitrogen into the distilling vessel containing a solution of 280 kg. of soda in 4,000 l. of water. After addition of 0.5 kg. of rape oil (or any other anti-frothing agent), the pyridine is vacuum distilled into an iron receiver at 30°-35°C. in 12-14 hr.

As soon as the distillation is finished, the contents of the vessel are filled up to 4,000 l., and after addition of 10 kg. of clarifying carbon, filtered at 80°C. through a wooden filter press pre-heated by steam to the iron salting-out vessel of 7.5 cu. m. The pressed product is rinsed with water of 60°C. and washed with warm water until anthrasol is no more detected in the filtrate. At first, the washing water runs to the mother liquor, then into the distilling vessel, where it is used for the next batch. Gradually, the filtrate is salted out with 10 per cent salt, cooled down while being stirred slowly and sucked off on the open iron suction filter. The filtrate runs into an iron receiver.

The product with 12 kg. of caustic soda solution (33 per cent) are converted into a paste in the iron stirring vessel of 0.5 cu. m.; then they are dried at 50°-60°C., at first under normal pressure, then in the vacuum. The product is 55 per cent pure. The dried product is ground on a gross beater mill and standardized against type (30 per cent of pure dyestuff) with Glauber's salt and 6-8 per cent of sodium dimethyl-anilinesulphonate.

In a special equipment, for drying, the pyridine, after addition of benzene, is azeotropically distilled from its aqueous solutions and redistilled. The loss amounts to 8-10 per cent.

*Process Control Tests* — (a) After finishing esterification, a sample from the esterification vessel is mixed with soda water, filtered at 60°C. and washed with warm water. The residue on the filter is tested by vatting as to absence of vat dyestuff.

(b) Before beginning of the distillation, a test is to be made as to phenolphthalein-alkaline reaction.

(c) The distillate is tested with acid and sodium nitrite as to a probable anthrasol content.

(d) The distillation is finished as soon as the pyridine content of the distillate is less than 1 per cent.

(e) Before clearing the ester solution, it must be ascertained whether the anthrasol is completely dissolved. If necessary, water has to be added.

(f) The pressed product is tested by extraction with hot water as to anthrasol, and by vatting as to vat dyestuff.

(g) A sample of the salted-out anthrasol is sucked off and the filtrate is tested with nitrite and acid for anthrasol.

*Raw Materials, Intermediates & Finished Product Specifications* — Vat dyestuff, pyridine and iron are to be tested by a test esterification in the laboratory.

In the finished product, the content of dyestuff is gravimetrically determined by analysis with iron chloride and acid. The yield is indicated as pure dyestuff of 100 per cent strength. A sample is standardized with Glauber's salt and 6-8 per cent of sodium dimethylaniline sulphonate to Anthrasol Brilliant Pink 13B powder and compared with the type by textile printing. Anthrasol Brilliant Pink 13B powder contains 30 per cent of the pure dyestuff. For testing the resistance to storing, a sample is kept for 3 weeks at 55°C. After that time it must not show any decomposition.

#### APPENDIX 61c

##### ANTHRASOL RED IFBB

*Principal Use* — For printing and dyeing cotton and artificial fibres.

*Equipment* — Iron vessel, 2 cu. m., with stirring device and jacket (esterification vessel); leaded iron vessel, 7 cu. m., with stirring device (acid separator); wooden suction filter with filter cloth, 3 sq. m.; iron vessel, 7.5 cu. m., with stirring device and jacket (distiller), with iron cooler and iron receiver; 2 wooden filter presses, 800×800 mm. iron vessel, 7.5 cu. m., with stirring device and jacket (vessel for salting out); and an iron vessel, 0.5 cu. m., with high-speed stirrer and lower outlet.

*Process* — In the esterification vessel 168 kg. of chlorosulphonic acid are run into 1,000 kg. of pyridine at 15°-20°C. within 3-4 hr. Then, 95 kg. of Indanthren Red FBB 100 per cent, dried and ground, and 100 kg. of bronze copper grindings are added at 23°-25°C. The temperature rises to 40°-43°C.; the esterification is mostly finished after half an hour. The batch is pressed by nitrogen into the acid separator which contains 4,000 l. of water of 10°C. After stirring for half an hour,

the pyridine salt of the ester becomes solid and granular; it is sucked off in the suction filter and washed with some water from the vessel.

In the distilling vessel the pyridine salt of the ester is introduced into 4,000 l. of water and 120 kg. of caustic soda solution (33 per cent) and after addition of 0.5 kg. of rape seed oil (or other anti-frothing agent), the pyridine is distilled under reduced pressure at 30°-35°C. into an iron receiver. When the distillation is finished, the vessel is filled up to 3,600-4,000 l. and filtered through a steam pre-heated wooden filter press into the vessel for salting out. The mass remaining on the filter is rinsed with water of 50°C. and washed until anthrasol is no longer detectable in the filtrate. The filtrate is salted out at 45°C. by addition of 10-12 per cent of salt (about 450 kg.) and cooled to 20°C. while stirring from time to time. The ester is filtered off in a wooden filter press and washed with about 1,000 l. of common salt solution.

The pressed product is pasted with 4 kg. of caustic soda solution (33 per cent), 32 kg. of molasses, 8 kg. of urea and 8 kg. of dimethylsulphanil salt in the vessel with high-speed stirrer and dried at 50°-60°C., at first under normal pressure and then under reduced pressure. The manufactured product has about 35 per cent purity and is adjusted to type with Glauber's salt after grinding in a cross beater.

The pyridine is azeotropically distilled. The loss amounts to 8-10 per cent.

*Process Control Tests* — (a) When the esterification is finished, a test portion taken from the esterification vessel is added to water and washed. The pyridine salt is dissolved in dilute caustic soda solution. On filtering only copper sludge, which is not coloured red by vat dyestuff, should remain.

(b) Before the beginning of the distillation a test as to reaction alkaline to phenolphthalein has to be carried out.

(c) The distillate is tested as to the absence of anthrasol by nitrite and acid.

(d) The distillation is finished when the pyridine content of the distillate is less than 1 per cent.

(e) Before clarifying, a sample is tested as to whether the anthrasol has been completely dissolved. If necessary, water has to be added.

(f) The residue remaining on the filter press is tested for anthrasol by extracting

with hot water and as to vat dyestuff by reducing.

*Raw Materials, Intermediates & Finished Product Specifications*—Vat dyestuff, pyridine and copper grinding has to be tested by test esterification in the laboratory.

The dyestuff content of the finished product is gravimetrically determined by means of iron chloride and acid. A sample is adjusted to Anthrasol Red IFBB powder by addition of Glauber's salt and compared with the type by textile printing. Anthrasol Red IFBB powder contains 30 per cent of pure dyestuff.

## APPENDIX 62

### TETRANITRO CARBAZOLE

*Principal Use*—Standardizing of Niro-san (plant protective agent).

#### *Equipment*—

(a) *Sulphonation*—Supply vessel for sulphuric acid, 12 cu. m. Sulphonation vessel, 5 cu. m., with stirrer and heating.

(b) *Nitration*—2 nitration vessels, 9 cu. m., with stirrer, 2 reservoirs, 20 cu. m.; 2 measuring vessels, 3 cu. m.

(c) *Effusion & Insulation*—5 effusion vessels, 12-20 cu. m., lined with Igelit and bricks, with stirrer and cooling coil; 2 admission vessels, 7.5 cu. m.; 2 filter presses 1,200 × 1,200 mm., 32 frames; 3 pasting vessels, 12 cu. m., with stirrer; 1 intermediate vessel, 7.5 cu. m., with stirrer; 1 vessel for dissolving sodium carbonate, 5 cu. m., with stirrer; 1 container for hot water, 5 cu. m.; 2 centrifugal pumps; 1 ventilator made of Igelit; 1 clay tower; 2 stoneware suction filters; and 2 electrical vacuum pumps, 15 kW.

(d) *Drying*—3 cylinder drying machines, heating surface, 23 sq. m.

(e) *Standardizing*—4 Perplex mills with mixing drum; 1 Excelsior mill with mixing drum; and 1 ventilator.

*Process*—Carbazole is dissolved in sulphuric acid, 95.5 per cent (formation of disulphonic acid and trisulphonic acid). Then it is mixed with nitrating acid containing 15 per cent of nitric acid and 85 per cent of oleum (20 per cent) and heated. The product is poured into water, filtered and washed. After conversion into a paste with water, the product is dried on a cylinder drying apparatus. The dried material is ground and mixed with bleaching clay.

*Process Control Tests*—Control of the strength of the sulphuric acid and of the nitrating acid.

Determination of the dry content of the tetranitrocarbazole and its nitrogen and sulphur contents. Determination of the sedimentation of a Niro-san suspension after 10 and 30 min.

*Raw Materials, Intermediates & Finished Product Specifications*—Carbazole is controlled by determination of its degree of purity. The efficiency of the finished products, tetranitrocarbazole and Niro-san, against pests is controlled in the plant protective laboratory.

## APPENDIX 63

### CARBAZOLE ESTIMATION ("IMIDO 1 ASE")

*Principle of the Method*—The carbazole is separated as N-nitrosocarbazole. It is then decomposed by boiling with ferrous sulphate solution and the nitric oxide produced is measured. Phenanthrene, anthracene and acridine do not interfere with the estimation.

For the quantitative estimation of carbazole, the determination of the m.p., solubility and chlorobenzene-insoluble substances are carried out. Besides, the anthracene content is also determined.

*Details of Analysis*—About 0.6 gm. (a) of the substance is dissolved by heating in a 300 c.c. Erlenmeyer flask with 40 c.c. glacial acetic acid using a reflux condenser. The solution is allowed to cool to 80°-90°C. and 30 c.c. of 0.5N sodium nitrite solution is added, the mixture is shaken and immediately placed in ice water. After frequent shaking and then letting it stand for 15 min., 150 c.c. of ice-cold water are added, and the mixture is allowed to remain in ice-cold water for a further 30 min. The separated nitroso-compound is carefully filtered on a filter funnel with paper filter. Any residue in the flask is brought on the filter paper with the help of the filtrates and washed 5 times with about 20 c.c. ice-cold water each time (test for nitric oxide).

The precipitate is again taken up in the flask with the help of 75 c.c. of pure glacial acetic acid and the flask is closed with a 3-holed rubber stopper carrying: (1) a tube provided with a glass stopper for passing carbon dioxide, the tube reaching the bottom of the flask; (2) a conducting bout tubing connected to an azotometer of 100 c.c.; and (3) a 50 c.c. dropping funnel. A strong current of carbon dioxide is passed through the flask till all the air is displaced which is established by allowing the gas

to collect over pure 30 per cent caustic potash solution in an azotometer and observing whether it is wholly absorbed.

The glass stopper of the tube is now closed and 25 c.c. of iron sulphate solution is added through the dropping funnel (200 gm. of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  plus 400 c.c. water plus 100 c.c. sulphuric acid), well shaken, and heated over a flame. In the end it is heated to boiling till the gas stream is completely absorbed. Now, for a short time, a moderate stream of carbon dioxide is passed through the flask which is again heated to boiling till all the gas stream is absorbed. The gas is allowed to remain over 30 per cent potassium hydroxide solution for 30 min., and its volume is measured. Simultaneously the temperature and pressure are determined. If  $b$  c.c. of nitric oxide, corrected to N.T.P., are obtained, the sample contains  $0.746 \frac{b}{a}$  plus 1.4 per cent carbazole, 1.4 being the solubility correction.

NOTE — If, instead of a commercial carbazole, a crude anthracene sample whose carbazole content is less than 30 per cent is taken for analysis, double the amount is weighed.

The solubility correction amounts to less than 1.4 per cent in the case of 15 per cent content and to 0.8 per cent in the case of 5-15 per cent content, and is very much lower in the case of 5 per cent content.

*Qualitative Tests* — (1) Determination of m.p.; (2) solubility determination.

7.5 gm. of 100 per cent according to analysis are dissolved by short heating in a 150 c.c. Erlenmeyer flask containing 100 c.c. of chlorobenzene. It is allowed to cool and remain overnight in a thermostat at 20°C. The precipitate is filtered in a weighed Gooch crucible, and the precipitate sticking to the walls of the flask is brought on the filter with the help of the filtrate, the precipitate is pressed well, washed with 25 c.c. benzene, dried and weighed. If the weight is  $c$  gm.

$$L = (7.5 - c) \times \frac{100}{7.5} = \text{solubility}$$

$$L \text{ for pure carbazole} = 8.8.$$

(3) Estimation of chlorobenzene insolubles: 2 gm. of the dried sample is dissolved by short heating in a 300 c.c. Erlenmeyer flask containing 100 c.c. of chlorobenzene, and provided with an air condenser. The hot solution is filtered in a weighed Gooch crucible, washed with pure 50 c.c. of

chlorobenzene at 70°-80°C. dried and weighed. If the weight is  $d$  gm.

$$d \times 50 = \text{per cent chlorobenzene}$$

insoluble matter.

*Estimation of Anthracene in Carbazole* — (a) *Gravimetric Estimation* — The gravimetric estimation of anthracene in carbazole is carried out with difficulty according to the method of Luck with the modification that the quantity of chromic acid solution is doubled.

(b) *Anthracene Estimation in Ultra-violet Light: Principle of the Method* — Anthracene in carbazole can be estimated with the help of an ultra-violet lamp based upon the strength of fluorescence by comparison with carbazole solution of known anthracene content. The fluorescence can be considerably intensified if the fluorescence due to carbazole is removed, i.e. if the carbazole is converted into a non-fluorescent derivative, while at the same time the fluorescence of anthracene is retained. This is easily accomplished by the conversion of carbazole into its nitro-derivative. This does not fluoresce and any disturbance due to the yellow colour of the nitroso-carbazole can be easily overcome by dilution.

*Details of the Determination* — 0.10 gm. of the carbazole under investigation is dissolved in 100 c.c. of glacial acetic acid in a 300 c.c. Erlenmeyer flask provided with a glass stopper, and slowly heated. The solution is allowed to cool, 1 c.c. of normal nitrite solution is added, the flask is closed, and allowed to stay for 10 min. and occasionally shaken. From this solution 5 c.c. are taken and diluted with 95 c.c. of glacial acetic acid, and its fluorescence is compared under an ultra-violet lamp with that of a solution of known concentration and known anthracene content.

## APPENDIX 64

### DIFLUORDICHLORMETHANE

*Principal Use* — Refrigerating agent.

*Equipment* — (1) 4 autoclaves, cast-steel lined with sheet iron, working pressure 50 atm. over normal; 2 of them with contents of 2,700 l. (1 of them in use, 1 in reserve); 1 of them with content of 250 l., and the other of 100 l.; (2) 2 Igelit washing towers filled up with Raschig belts of graphite; (3) 2 iron washing towers; (4) 1 iron gas container, contents of 6 cu. m.; (5) 1 compressor (capacity per hr. 40 cu. m., maximal pressure 20 atm. excess pressure);

(6) 1 iron supply vessel for crude Frigen, content of 2 cu. m., working pressure of 13 atm. over normal; (7) 1 iron supply vessel for concentrated hydrofluoric acid, contents of 20 cu. m., working pressure of 3.5 atm. over normal; (8) 1 iron weighing vessel for concentrated hydrofluoric acid, contents of 0.5 cu. m., working pressure of 3.5 atm. over normal; (9) 1 iron supply vessel for carbon tetrachloride, contents of 20 cu. m.; (10) 1 iron supply vessel for sulphuric acid, contents of 10 cu. m.; (11) 1 iron supply vessel for caustic soda solution, contents of 10 cu. m.; (12) 1 iron distilling vessel for Frigen, contents of 2 cu. m., working pressure of 2 atm. over normal while working with column; (13) 1 iron receiver for first running; (14) 3 iron receivers for pure Frigen, contents of 5.5 cu. m. each, working pressure of 13 atm. over normal; (15) iron vessel for crude  $F_{11}$ , contents of 0.9 cu. m., working pressure of 3.2 atm. excess pressure; (16) iron vessel for pure  $F_{11}$ , contents of 0.9 cu. m., working pressure of 3.2 atm. over normal; (17) iron distilling vessel for  $F_{11}$ , contents of 0.8 cu. m., working pressure of 3.2 atm. over normal while working with column.

*Process* — The autoclave (1) is charged with 400 kg. of antimony trichloride. Then, out of a measuring vessel, 1,540 kg. of carbon tetrachloride, in which about 20 kg. of chlorine are dissolved, are run in (1). Finally 500 kg. of hydrofluoric acid (98-99 per cent) are weighed in (8) and pressed into (1) by compressed air. The gas is allowed to blow off the autoclave which is heated with low pressure steam at about 30 atm. Control through a baffle plate with a differential manometer. Finally the autoclave is entirely released and cooled down. In (2) the released gas is set free from the main bulk and, in a washing tower (3), sprayed with caustic soda solution from the last traces of hydrochloric and hydrofluoric acid. The gas collects in (4), is dried in a washing tower (3), which is sprayed with concentrated sulphuric acid, and liquefied to crude Frigen through (5) with a brine, condenser fixed behind. During this process Frigen and inert gases, containing eventually  $F_{11}$ , are allowed to blow off through a release valve.

The cooled-down autoclave is filled up once more. The catalyst, i.e. antimony chloride, remains in the autoclave until exhaustion.

The crude Frigen yield of one operation (about 1,200 l.) and the first runnings of a previous distillation (about 200 l.) are drawn off into the distilling vessel (12); here they are fractionated in the column with little steam (steam jacket), while beginning with depriving of air up to the constancy in pressure and, after having taken off first runnings (about 200 l.), the pure Frigen is collected in one of the receivers (14). The residue in (12), 70-90 l., is, after the release, still crude  $F_{11}$  contaminated with Frigen and higher-boiling constituents, and is pressed to (15). This crude  $F_{11}$  either goes back to the manufacturing process instead of carbon tetrachloride or is fractionated in (17), where pure  $F_{11}$  is collected in (16). The development of an additional apparatus for the technical manufacture of  $F_{13}$  is still in its beginnings. According to the above-mentioned method the by-product  $F_{13}$  results only in a small quantity, so that its manufacture is not paying.

Yield of pure Frigen = 1,090 kg.

*Process Control Tests: Examination of Frigen* —

(a) *Determination of Water* — 300 l. Frigen gas, measured with a gas meter, are conducted through 3 weight tubes filled with phosphorus pentoxide. The increase in weight gives the content of water. The percentage of water in the Frigen

$$\frac{\text{increase of weight}}{750} = 100$$

(b) *Constituents Insoluble in "Bayolol"* — 100 c.c. Frigen gas are taken off with a Hempel mercury burette. This burette is connected with a Hempel absorption pipette filled with "Bayolol", in which the gas is shaken up to the constancy of volume of insoluble gas determined in the burette.

(c) *Boiling Interval & Higher-boiling Contaminations* — A little more than 100 c.c. liquid Frigen are put into a Dewar vessel with graduations, the Frigen is evaporated in the presence of several carborundum crystals and the boiling temperature is determined from 5 to 5 c.c.

(d) By adding 5 c.c. of Frigen to a solution of silver nitrate in 5 c.c. of absolute alcohol, the solution should not be rendered turbid.

*Raw Material, Intermediates & Finished Product Specifications* — (a) The first boiling point of Frigen near 760 Torr must not be higher than  $-29^{\circ}\text{C}$ . The boiling

interval between the first boiling point and the point when only 15 per cent of the sample are left, must not exceed 0.5°C. Frigen must not contain more than 0.0025 per cent water. Constituents insoluble in "Bayoloil" must not exceed 5 per cent by volume. Frigen must be entirely neutral (free from hydrochloric acid).

(b) The hydrofluoric acid must be of a strength of more than 96 per cent, if possible, 98-99 per cent.

#### APPENDIX 65

##### OCTADECYL-ISOCYANATE (Intermediate for Persistol VS)

*Principal Use* — For producing water-repelling effects on fibres.

*Equipment* — 3 enamelled vessels, 1 cu. m. each; 2 homogeneously leaded receivers, 500 l. each; 1 iron distilling vessel with receiver, 500 l.; and 1 equipment for destroying phosgene.

*Process* — Into the 1 cu. m. enamelled vessel 300 kg. of chlorobenzene are filled, heated to 60°C. and 300 kg. of octadecylamine, previously melted, are added. The temperature is raised to 90°C. and the amine transformed into its hydrochloride by introducing about 40 kg. of hydrochloric acid gas. The batch is next pressed into the second enamelled vessel and, at a

temperature of 105°-110°C., 10-15 kg. of phosgene per hour are next introduced. The total quantity for the conversion into the isocyanate is about 150 kg. of phosgene.

After stirring for 2 hr., nitrogen is passed through the batch for 3-4 hr. in order to expel hydrochloric acid and phosgene and then the chlorobenzene is distilled off *in vacuo* (about 100 mm.). Temperature can be raised up to 160°C. The residual raw isocyanate is distilled *in vacuo* (3-5 mm.) at a temperature of about 210°C. to the pure product. The unreacted phosgene, escaping together with hydrochloric acid, is destroyed in an absorption plant.

Yield from one batch: about 280 kg.

*Process Control Tests* — The final point of the phosgenation is determined by distillation of a sample. There must not be more than 5-6 per cent of undistillable residue.

The progress of the conversion is recognizable by the fact that in a cooled sample the quantity of octadecylamine-hydrochloride, insoluble in chlorobenzene, decreases more and more, a clear, brown solution finally resulting.

*Raw Materials, Intermediates & Finished Product Specifications* — Octadecylamine: the raw product of about 90 per cent is previously distilled *in vacuo* to the pure product. The finished product is a clear, transparent oil.

(To be continued)

## Quality of Sugar Manufactured in India

THE DIRECTOR, INDIAN INSTITUTE OF SUGAR Technology, Kanpur (India), has issued his review on the quality of sugar manufactured in India by the central sugar factories and refineries for the season 1947-48.

The data supplied by 124 factories show that there is no significant improvement in the quality of sugar manufactured in 1947-48 over the sugar of the previous season. In fact, the production of ISS. No. 28, which had occupied a position next to the standard ISS. No. 27, has registered a drop from 23.28 per cent in 1946-47 to 17.76 per cent in 1947-48. As before, the most predominant colour grade in 1947-48 was ISS. No. 27, which accounted for 56.33 per cent of the total production. The production of ISS.

No. 26 was slightly higher. 98 per cent of the total production was in the colour grades ISS. Nos. 28, 27, 26 and 25.

Regarding grain size there was a reduction in size A and size C, while a corresponding increase in size E was recorded. Over 97 per cent of the total production was covered by the sizes C, D, E and F, the size D alone accounting for 47.68 per cent.

What is disappointing is that some factories produced a large number of grades of sugar to the extent that one factory recorded 23 different grades. It is highly desirable that methods of standardization should be strictly adhered to so that the number of grades produced by any factory is restricted.

G. G. RAO



# REVIEWS

**Waste-heat Recovery from Industrial Furnaces**, being a treatise based upon a series of papers presented to the *Institute of Fuel* (Chapman & Hall Ltd., London), 1948, pp. 384+ix. Price 35s. net.

THE VOLUME UNDER REVIEW CONSISTS OF A number of papers by different authors. The papers were presented in the first place to the *Institute of Fuel*, London, and owe their origin to the urgent necessity during war-time to conserve the fuel of Great Britain. They are all concerned with the study of heat transference and heat insulation and the recovery of waste heat from industrial furnaces with the object of increased overall efficiency. The Committee of the *Institute* responsible for the symposium is to be congratulated on presenting an important and useful treatise which, in spite of its mixed origin, gives the impression, not of piecemeal or patch-work composition, but of a coherent volume. This is attributable no doubt to able and careful editing.

The papers or chapters carry the following headings: (i) Total, Recoverable, and Returnable Heat in Combustion Gases by P. O. Rosin; (ii) Some Simplified Heat Transfer Data by Margaret Fishenden and O. A. Saunders; (iii) An Experimental Determination of the Factors Governing the Design of Regenerators with special reference to Coke-ovens by T. C. Finlayson and A. Taylor; (iv) Tubular Metallic Recuperators by G. N. Critchley and H. R. Fehling; (v) Waste-heat Boilers, by W. Gregson; (vi) The Utilization of Waste Heat in the Carbonizing Industries by J. G. King and F. J. Dent; (vii) Waste-heat Recovery in the Metallurgical Industry by J. A. Kilby, W. G. Cameron, E. C. Evans, A. H. Leckie, J. L. Harvey and A. E. Balfour; (viii) Waste-heat Recovery in the Glass-container Industry by W. A. Moorshead.

There is a brief but pertinent introduction by G. N. Critchley, showing in striking figures the money-value of the fuel saved by installing fuel-saving (i.e. waste-heat recovery) equipment. The cost of the equipment is stated. There is an excellent index.

In the body of the book the main industrial furnaces considered are those dealing with

coal carbonization, the metallurgical industries and the glass industries. The first two chapters are devoted to basic fundamental considerations; and they are the more important as Rosin's treatment of "Total, Recoverable, and Returnable Heat in Combustion Gases", and the simplified "Heat Transfer Data" of Fishenden and Saunders are often referred to in the subsequent portions of the book. It is consequently advisable for the serious readers to spend time on these early chapters. They comprise only 66 pages.

Rosin's paper is an attempt at simplification of the usually tedious calculations involved in working out combustion and heat losses: it should be read with care. Perhaps its main service will be to help the technologist to visualize roughly but rapidly the prevailing combustion and heat conditions. Rosin's work is seldom easy reading — and this chapter is (to the reviewer) no exception. At the outset we come across an irritating statement. "Combustion", he says, "is an industrial process for the manufacture (*sic*) of hot gases which are to be used in another industrial process for the production of goods of various kinds, such as metals, ceramics, coke, or chemicals." While a definition must delimit, it must also embrace all that it should — and to define combustion in the above terms is simply not true. If we are dealing with a particular *aspect* of combustion, we should say so. But perhaps the main objection to the statement is that by the end of the sentence we have lost sight of the combustion process itself and are dabbling in "metals, ceramics, coke, or chemicals".

Rosin in this chapter spends time on developing a general or "statistical" relation between the net calorific value, the air requirement and the combustion gas volume for different fuels, and he deduces useful approximations. Perhaps the *most* useful is that the heat content of unit volume of the combustion gases obtained with theoretical combustion is roughly constant and independent of the calorific value of the fuel.

A later portion of the chapter is devoted to the heat content of combustion and waste gases and to the I/t diagram. The substance



of this section requires care from the reader by way of interpretation, and the same remarks apply to the last section on "Recoverable and Returnable Heat". Chapter I is of basic importance and contains much valuable matter; but it is perhaps not written with the clarity which it deserves.

Chapter II by Fishenden and Saunders is a competent statement on the elements of heat transfer. It contains a brief treatment of the "numbers" of Reynolds, Prandtl (Stanton), and Nusselt. The complex nature of heat transmission is indicated, and there is a brief explanation of heat transfer by radiation and by forced and natural convection. The usefulness of this contribution is enhanced by the heat transfer data assembled in the last few pages of the book.

Finlayson and Taylor in the "Design of Regenerators with special reference to Coke-ovens" have written an essentially practical essay of value to coke-oven manufacturers and all concerned with design in this field. "Tubular Metallic Recuperators" by Critchley and Fehling embraces practical, experimental, and theoretical aspects of its subject. Corrosion is not overlooked, but the treatment here is none too thorough.

W. Gregson's chapter on "Waste-heat Boilers" is a general review, largely descriptive, and contains little revolutionary except a plea for the use of the gas turbine. Nevertheless, it constitutes a balanced statement in the body of the book: it will be valued more especially by the non-technical reader.

In "The Utilization of Waste Heat in the Carbonizing Industries", King and Dent give a comprehensive description of their investigations into waste heat in coal carbonization practice. The improvements possible with horizontal gas retorts, continuous vertical retorts, intermittent vertical chambers, coke ovens, and water-gas plant are indicated. The essay is lengthy, but will repay careful attention by all concerned with the manufacture of coal gas or water gas. The brief appendix by Dr. S. Pexton is an admirable statement on the balance of fuel, steam and power in carbonizing plants.

Chapter VII, by several authors, deals with "Waste-heat Recovery in the Metallurgical Industry". A large section is devoted to the iron and steel industry including coke-oven plant, blast furnaces and cupolas, melting furnaces, and reheating and heat-treatment furnaces. The dry-cooling of coke receives full and reasoned consideration. The second "half" of the chapter is devoted to the non-

ferrous metal industry, and the future development of waste-heat boilers in connection with metallurgical industries. Reference is again made to the driving of gas-turbines by means of high pressure waste gas from boilers burning compressed blast-furnace gas and air.

In "Waste-heat Recovery in the Glass-container Industry", W. A. Moorshead shows the application of the theoretical principles enumerated in chapters I and II to the problems of the glass industry. The case for the use of waste-heat boilers with glass-melting furnaces is so strong as barely to need argument.

The book, therefore, constitutes a good general guide to modern practice in the recovery of waste heat from industrial furnaces. The usual line of procedure at works is to utilize the sensible heat in the hot gases either for steam raising or for heating the air to be supplied for the combustion process, employing in the latter case regenerators or recuperators. Mention has already been made of a further possible development, namely the utilization of the hot gases in gas-turbines.

The problem of waste heat, however, exists in rather larger context. Waste heat may result not only from hot flue gases, but also from incompletely burnt fuel or hot rejects in any form. The present treatise leaves one with the feeling that a first-class comprehensive volume on the utilization and/or reduction of waste heat still remains to be written. The treatment of fundamentals given in the present volume certainly needs expanding to cover the field. But this expansion must not be cumbersome and at the expense of clarity. The subject is an intensely practical one, and the book must have its appeal essentially to engineers of all ranks. Carbon balances, however, must not be overlooked, and the losses due to incomplete combustion or to rejection of hot fuel or even hot solid or liquid matter from furnaces should be considered. No doubt we have stepped outside the field envisaged as "Waste Heat", but the line of demarcation is artificial.

It remains to say that the appearance and printing of the book are good and that in its present form it will be of great service to engineers and fuel technologists concerned with waste-heat recovery. But may we not ask the *Institute of Fuel* to use it as a first step towards the compilation of further treatises on fuel economy?

J. W. W.

**Television Production Problems**, by J. F. Royal (NBC-Columbia University Broadcasting Series) (McGraw-Hill Book Co. Inc., New York & London), 1948, pp. xii+179. Price \$2.50.

THE COLUMBIA UNIVERSITY IN CO-OPERATION with the National Broadcasting Company started a scheme of instruction in all phases of radio programming and operation with as much workshop experience as possible. This book is based on an extension of the scheme to the art of television. It is really a course of lectures delivered by different authors touched up by the editor.

The following are the main topics dealt with: (i) Introduction by J. F. Royal; (ii) The Science of Television by F. A. Wankel; (iii) Television Writing Problems by R. P. McDonough; (iv) Television Production Facilities by N. R. Kelly; (v) Settings for Television by R. J. Wade; (vi) Production of Dramatic and Variety Programs by E. Sobol; (vii) Opera in Television by H. Graf; (viii) Problems in the Studio by F. Coe; (ix) Television Mobile Unit Broadcasting by B. Crotty; (x) Legal Problems in Telecasting by I. E. Lambert; and (xi) Television Advertising by R. Kraft.

The above "titles" indicate clearly the wide variety of topics dealt with. All the authors are experienced persons engaged by the NBC. The book covers a complete course of instruction that need be given in this field and is *typically* American. We do not believe yet in this type of courses in the universities. In the States there is considerable demand for specialization and the book is a clear indication of the way in which the Americans feel the need. Television is yet to make its way into India and when it comes in, the book will be most useful to the staff-training schools. But there is another way in which the book can find its utility. It is a very valuable addition to the general section of college libraries and as a book for general reading to our communication engineering students. As one reads through the book, one finds little that is new and there is little that does not occur as a matter of course to a systematic thinker. But we must remember that originality consists not in thinking differently from others but in thinking for oneself!

The printing and get-up of the book are excellent. The numerous photographs are aptly chosen and beautifully reproduced. The book can as well go in as a model

to those engaged in book production problems!

S. V. CHANDRASHEKHAR AIYA

**An Introduction to Comparative Biochemistry**, by Earnest Baldwin (Cambridge University Press), 1948, 3rd edition, pp. xiii+164. Price 7s. 6d.

THIS EXCELLENT LITTLE VOLUME NEEDS NO introduction to students of biochemistry. The fact that the book has gone through 3 editions (once reprinted) in the course of a decade bears ample testimony to its excellence and to the popularity which it enjoys with those interested in biochemistry.

The author states that the aim of the book is twofold: first, to provide an elementary text-book of this special subject suitable for students of biochemistry, and, secondly, to provide a starting-point for those who, for any reason, may find themselves attracted to the subject. These two objectives have been more than fulfilled in the short space of 164 pages. The subject-matter has been selected carefully and the exposition is lucid. As Professor Hopkins has stated, the author has "illustrated very adequately, in relatively few pages, many of the main lines of current progress, leaving in the mind of the reader no doubts concerning their significance. Above all, he has displayed his own interest in the subject, an interest, intense enough to be infectious."

This edition has been revised, considerably enlarged, re-set and brought up to date. A new section on the transport of carbon dioxide has been added to chapter VI, and a new chapter (VIII) has been introduced to cover the comparative aspects of nutrition, digestion and metabolism, adding materially to the usefulness of the book.

A number of useful books and reviews has been added to the list of references. A classification chart is included to provide the student, who is not familiar with zoological nomenclature, with a ready means of deciding to what phylum or class a given animal should be assigned.

A. K.

**Supersonic Flow and Shock Waves**, by R. Courant & K. O. Friedrichs (Interscience Publishers Inc., New York & London), 1948, pp. xvi+464. Price \$7.00.

THIS BOOK IS THE FIRST OF THE SERIES OF books and monographs on pure and applied

mathematics edited by H. Bohr, R. Courant and J. J. Stoker. The first two names are familiar to all students of mathematics, the latter of them being the author of the celebrated *Methoden*. The book under review appears to be an off-shoot of the scientific work done in connection with the last war and is, therefore, relevant to problems of national defence. The book is a model of clear and accurate exposition of a mathematical subject.

The book consists of six chapters. In the first the fundamental equations of hydrodynamic flow with proper thermodynamic considerations, but omitting viscosity and heat conduction, are obtained. In the second the differential equations of the hyperbolic type involving two variables are thoroughly discussed by the help of characteristic curves. In the initial value problem, the concepts of domain of dependence and range of influence are introduced. Propagation of discontinuities along characteristic lines is then considered and a discussion of the hodograph transformation is given. Generalizations to more than two variables are indicated.

In the third chapter specific problems of one dimensional flow are taken up. Shocks are regarded as irreversible processes described by sudden jump discontinuities occurring across sharply defined surfaces. The jump conditions and theorems relating to shock transitions are established. Treatment is given of reflection of a shock by a rigid wall, interaction of a shock, Riemann's problem, detonation and deflagration waves, Chapman-Jouguet processes, Jouguet's rule and wave propagation in elastic-plastic material.

In the fourth chapter the treatment of special flows by the hodograph method is given, characteristics, Mach lines and Mach angle are introduced and then follow flows round a bend, along a bump and in a two-dimensional duct, phenomena in a jet by gas streaming in parallel supersonic flow out of an orifice and an account of shock polars, oblique shocks and flow round an air foil by perturbation methods. The boundary conditions for steady flow are critically examined and scope for further work is indicated. A qualitative discussion of flow in nozzles and jets follows in the fifth chapter. Cylindrically symmetric steady flow, steady conical flow and non-steady flow with spherical or cylindrical symmetry are treated in the last chapter of the book.

An excellent comprehensive bibliography is given at the end of the book. But the

regret is that some of the important references come under unpublished war work. References to Chandrasekhar, the distinguished astrophysicist, Bethe and Gamow, the distinguished nuclear physicist and Weyl and Neumann, the famous top-ranking mathematicians, are found in this work. Chandrasekhar's reports to the Aberdeen Proving Ground on the normal reflection of a blast wave, on the decay of plane shock waves and on the condition for the existence of three shock waves have been referred to in the bibliography. This shows the amount of interest taken by the leading mathematicians and physicists on problems relating to national defence in a time of emergency.

N. S. NAGENDRA NATH

**Electrical Accidents**, by K. V. Karantha (Harsha Printery & Publications, Puttur, S. Kanara, Madras), 1948, pp. iii+136. Price Rs. 3/8.

THE BOOK IS THE FIRST OF ITS KIND IN INDIA. The subject-matter is dealt with in great detail with a number of illustrations from practice which can be understood by a reader with a preliminary knowledge of electricity. The book is written from the author's own experience and is based on the general practice followed in the province of Madras. It would have been better if information about the practice in other provinces was also incorporated and it is hoped that this will be done in the future editions to enhance its usefulness and make it a standard book of reference all over India.

The author has given in detail the causes of accidents in houses and outside, and has suggested the enforcement of Rule 48 of the I.E. Rules, 1937. In the Bombay province, the statutory provisions regarding the electric installations in houses, factories, places of public entertainment, etc., are strictly enforced and a licencing board grants licences to electrical contractors who are authorized to take up such works and conducts examinations for electricians and wiremen. It would be interesting to know the methods adopted in other parts of India. If the regulations in force have not actually prevented accidents, they will have to be modified in the interest of safety to the public and extension of electric power to rural areas.

The author has clearly described the physiological effects of the passage of electric current through the human body and explained the circumstances under which the

electric shock is likely to prove injurious and fatal. The chapter regarding artificial respiration should have followed this, and it should have included other methods such as mechanical respiration aids. Recommendations of a few medicines commonly used in cases of electric burns would have been useful.

The chapter on the causes of electric accidents in dwelling houses, factories and in public utility concerns are very exhaustive and show conclusively how most of these accidents could be avoided by proper observance of the prescribed regulations under the *Indian Electricity Act and Rules*. The author has given details of clearances from the live conductors and the safety measures such as fencing, anti-climbing devices, etc., prescribed by the Madras Government. It would be better if these are standardized on an all-India basis and prescribed under the authority of the Central Electricity Board.

Provision of an adequate "earth" being essential for the operation of all safety devices such as fuses or earth leakage relays of circuit breakers, etc., it is very essential that this question should be properly understood. The author has considered this aspect and described in detail as to how proper earth connections should be made and how very low earth resistances should be maintained. This information would be very useful for all public utility engineers and engineering contractors.

The author has devoted a special chapter for the multiple earth neutral system and has shown both the advantages and disadvantages of the same and its application in the Madras province. The adoption of this system would be very useful for other provinces.

The causes of fire accidents by the use of electric appliances in houses and factories are discussed in detail and helpful suggestions to avoid them are given. Discussion and implications of some extracts from the regulations for the installation and maintenance of electric installations prescribed by the "fire insurance companies" would be very useful and should be included as an appendix.

On the whole, the book is very instructive and useful as a text-book for technical institutions and for examinations conducted by the Provincial Licencing Boards and as a reference book for the public utility concerns and factories. The author must be congratulated for publishing such a valuable book.

B. K. R. PRASAD

**The Indian Sugar Industry, 1947-48, Annual**, edited by N. P. Gandhi (Gandhi & Co., Bombay), 1948, Vol. XIII, pp. xlviii+136+32. Price Rs. 6.

THE PRESENT ANNUAL REVIEW OF INDIAN Sugar Industry, like its fore-runners, contains an exhaustive account of the progress of the sugar industry during 1947-48, all the relevant data of the previous years being brought into well-compiled tables for ready comparison.

The author's preface is a succinct account of the present problems of the sugar industry and his own views on them. There are 41 highly informative tables given at the very beginning of the book. The review covers the progress of not only the sugar industry, but also the other allied subjects like power alcohol and cane cultivation. The subject is dealt with in all its aspects, and the world position is reviewed. An appendix giving an up-to-date list of sugar mills of India and Pakistan is included.

The author lays special stress on the futility of an elaborate tariff enquiry on the continuance of protection to sugar at a time when the industrial conditions, both in India and abroad, are very unstable, costs of production violently fluctuating, and the future of production, consumption and world trade in sugar cannot be predicted owing to the prevalence of insecure conditions. Protection to sugar industry must be maintained at the present juncture in the interest of the agricultural economy of India. The author has stressed the importance of intensified agricultural research and, therefore, the necessity of restoring the grant agreed to by the Government of India in 1947. This grant is estimated to be about Rs. 50 lakhs per year. A wider sales organization, on the same lines as of NIWAS of Java to cover every factory in India, has been advocated. Among other points stressed by the author are: improving the manufacture of *gur* and encouraging the manufacture of palm *gur* and the necessity for reducing the price of sugar by reducing the price of cane and the sugarcane cess to give an impetus to increased production and consumption of sugar.

The Annual cannot be missed by anyone closely associated with, or interested in, the Indian sugar industry.

G. G. RAO

**Plants Diseases : Their Causes and Control**, by Sudhir Chowdhury (Kitabistan, Allahabad & Karachi), 1948, pp. 106. Price Rs. 4/8.

AS ONE GOES THROUGH THIS LITTLE VOLUME, the impression that the book has not been planned properly and that the subject-matter has been inadequately and poorly treated becomes confirmed by the time the end is reached. The author states that at the request of his students he has published his lectures in the form of a booklet. It may be that in the form of lectures the subject-matter was interesting enough to students, but, to publish the material in the form of a book, the author should have given greater attention to precision in terminology and proper sequence in the presentation of the subject.

The book is divided into six chapters. After a short introduction (which forms chapter I), "definitions and symptoms of diseases" are discussed in the second chapter. The material is evidently based on Heald's Manual of plant diseases, and the author's attempt at brevity and simplicity has resulted in a large number of inaccurate statements and generalizations which, in the opinion of the reviewer, is not beneficial to students for whom the book is intended. Chapter III should have formed the introduction as it is very necessary for a beginner to know the structure, life history, etc., of the causal organisms before making an attempt to study the diseases they produce and the symptoms which are manifested.

The book abounds in grammatical, spelling and typographical errors. There is little in the volume which the reviewer can commend to readers. The publication is priced high; a third of the price would have been reasonable.

V. S. RAO

**German Primer for Science Students**, by Haragopal Biswas (University of Calcutta), 1948, 2nd edition, pp. xiii+250+i. Price Rs. 7/8.

THIS PUBLICATION IS FAMILIAR TO STUDENTS in this country, and particularly to those who have attempted to gain a working knowledge of scientific German by the "self-taught" method. The reviewer himself had occasion to use the book (1st edition), and he can unhesitatingly recommend it as a useful aid to the learning of German.

The present edition has been revised and a number of fresh exercises have been added. The sub-sections of exercises in physics, physical geography, pharmacology, chemistry and mathematics have been enlarged. An interesting feature of the new edition is the inclusion, in the last chapter, of passages of general scientific interest, biographical sketches of men of science, and a selection of well-known Indian short stories and extracts from the works of Kalidasa, Goethe, Shakespeare, Schiller and Tagore.

A. K.

**Elastomers and Plastomers, Their Chemistry, Physics and Technology, Part III**, edited by R. Houwink (Elsevier Publishing Co. Ltd., New York, Amsterdam, London, Brussels; Cleaver-Hume Press Ltd., London), 1948, pp. 174. Price 25s. net.

THE PUBLICATION OF PART III BEFORE PARTS I and II of the volume on elastomers and plastomers might appear a bit unusual as would, for instance, serving first the last dish of a meal while the others are still in preparation. But the reason for it might very well be, as in the case of Elsevier's 20 volume *Encyclopedia of Organic Chemistry*, whose publication started off with the 14th volume, the practical consideration of immediate public demand for the sort of information contained in this particular part. In fact, whereas parts I and II are to be entitled, respectively, "General Theory" and "Manufacturing Properties and Applications", part III, under review, deals with the methods of testing and analysis of elastomers and plastomers in a manner which will be much appreciated by those who have had up to now to refer for such information to scattered publications in journals and to the *American Society of Testing Materials (A.S.T.M.) Standards*, the *British Standard Specifications (B.S.S.)*, or even the *German Verband Deutscher Elektrotechniker (V.D.E.)*, among which, however, there has not been much uniformity in terminology or test specifications.

Chapter 1 is a page and a half introduction by Dr. R. Houwink of Wassenaar (Netherlands) on the general nature and significance of the physical and chemical tests and numerical data presented in the succeeding chapters. Dr. J. H. Teeple of *Celenese Corporation of America* treats, in chapter 2, the standard methods of testing the mechanical

thermal, optical, chemical and electrical properties of plastics and rubbery materials, explaining clearly and concisely the scientific principles, specific test conditions and inherent limitations involved in each type of tests and furnishing diagrams of testing machinery and tables of typical experimental data. This chapter is an excellent introduction and a helpful guide to the use of the American, British and German standard specifications of individual plastomers and elastomers.

Chapter 3, of particular interest even to practised organic chemists not well-versed in the analysis of high polymeric materials, is contributed by Dr. A. G. Epprecht of Zurich, in whose laboratory several of the analytical methods were either modified or developed. Starting with the method of preparing the material for analysis, since any given plastomer or elastomer consists of many other ingredients than the basic resin or latex, the author details successively the procedure for preliminary examination, systematic analysis along with confirmatory tests and a scheme of qualitative analysis of the more common plastics and rubbers, based on their solubility in organic solvents and resistance to both organic and inorganic chemicals, and finally gives several examples of quantitative analysis of individual polymers and plasticizers. Chapter 4 by Dr. B. B. S. T. Boonstra of the *Rubber Foundation*, Delft (Netherlands), contains, in tabular form, valuable numerical data of the properties of elastomers, a large part of which was obtained in his own laboratories, with explanations of the terms and standards prevalent in U.S.A. and other countries. Chapter 5 by J. W. F. van't Wout of Delft and Dr. R. Houwink presents similar tables of numerical data of the properties of plastomers. A detailed table of contents and a comprehensive index complete the volume.

Written by competent authorities and comprising within a comparatively short space a great deal of information on elastomers, the book is recommended, without reserve, to research workers in high polymers. The printing is good and no typographical mistakes were noticed. The only complaint which could be made is that the price is a little forbidding, almost two pence a page, although books of this type are nowadays generally expensive.

L. M. YEDDANAPALLI

**Plasticity as a Factor in the Design of Dense Bituminous Road Carpets**, by L. W. Nijboer (Elsevier Publishing Co. Ltd., New York; Cleaver-Hume Press Ltd., London), 1948, pp. xv+184. Price 28s. net.

AMONG THE ROAD MATERIALS IN USE, BITUMINOUS road mixtures play an important rôle in the development of roads designed to meet the exacting demands of modern traffic with respect to load, speed and safety. For gauging the potentialities of this road material and putting it to the best use, it is essential to have a detailed knowledge of its mechanical properties.

This book deals with methods of investigating the plastic properties of bituminous road materials and the practical applications of bituminous compositions.

The subject-matter is divided into 8 chapters. Chapter I gives a survey of road-building methods through the ages with particular stress on bituminous roads and functions and properties of bitumen. Properties of bituminous mixtures are studied, their mechanical properties analysed and existing testing methods reviewed.

Chapter II deals with the theory of the triaxial shear test method, testing apparatus and preparation of test specimens. The validity of the theory and the accuracy of the method are discussed, and a clear idea is presented of the resistance of bitumin-aggregate mixture to plastic flow as indicated by the 3 physical constants: coefficient of friction, viscosity and initial resistance.

Chapter III on mechanical properties at plastic deformation includes the quantitative assessment of the influence of changes on the composition and properties of mixtures subjected to different test conditions. The comparison of properties under standard conditions is thus rendered possible and the properties of mixes of variable composition can be evaluated when the properties of the standard mix are given. This is an important practical aspect of the subject, as mixes can be designed to meet the traffic requirements of the road.

In chapters IV and V, bearing capacity and compaction by rolling are discussed from the theoretical and experimental viewpoints, and the test apparatus specially developed for these investigations are described.

Chapters VI and VII contain tables and graphs relating to the grading and design of mixtures. These are of great help to research engineers.

In chapter VIII are to be found many observations on mechanical properties of bituminous road mixtures—a new line of approach based on the principles of soil mechanics. This has resulted in the clarification of the rôle of many variables in the bituminous road mixtures. The results obtained are not conclusive enough to formulate specifications for the composition of mixtures to meet any given requirement. These results have, however, led to the conclusion that road carpets might be designed to possess a "Prandtl Bearing Capacity" equal to the inflation pressure of pneumatic tyres. This study was restricted to hot mixtures, and research on cut-back mixtures and emulsion mixtures have still to be carried out.

The author's endeavour has been to bring the knowledge on mechanical properties of bituminous road materials to a level comparable to that of steel and cement concrete in order to obtain the same degree of safety in construction. In this attempt he has succeeded to a great extent, and this book will be of much use both to research workers and field engineers engaged in the construction of bituminous roads.

R. K. N. IYENGAR

**Photography in Crime Detection**, by J. A. Radley (Chapman & Hall Ltd. London), 1948, pp. 186. Price 21s.

CRIME PLAYS, AND HAS ALWAYS PLAYED, AN important rôle in the life of mankind since time immemorial. In the current century, it is said, crime has become a refined scientific pursuit. Those who fight against crime are, therefore, facing the need for utilizing all the advances of modern science for detection and prevention of crime.

In this book, *Photography in Crime Detection*, the author has managed to pack, within 186 pages, a staggering amount of information on the various uses of modern photographic processes in detection, analysis and proof of crimes. While keeping within limits of popular appeal, the author has made the chapters intensely practical and valuable alike to the working detective or

legal adviser as to the general reader who finds interest and diversion in greedily imbibing detective fiction.

After a theoretical introduction concerning the rôle of photography in crime detection, the author goes on to discuss cases involving mechanical vehicles, identification of persons, criminal cases such as murder, house-breaking, counterfeiting coins, gems and precious stones and betting offences. The examination of documents which are disputed on the ground of forgery and the photographing of such documents by normal and oblique lighting for purposes of comparing handwriting, identification of inks, detecting alterations, erasures or additions, etc., are dealt with in detail in a long chapter covering nearly 30 pages. The use of ultra-violet light and fluorescence analysis of documents, photographing documents, fingerprints, etc., by fluorescent light, ultra-violet light and infra-red light are covered in a very practical manner under separate chapter headings. The place of photomicrography and X-ray photography in crime detection is rightly emphasized and exhaustive practical information given under relevant chapter heads.

On the whole the book is an admirable condensation of very useful and important information on all aspects of the subject, and at the end of each chapter there is a bibliography which helps the reader to refer to other literature for additional information.

No less than 104 reproductions of photographs, actually used in various criminal cases, illustrate the book.

The author deserves special commendation in keeping one main principle in view which he states in his preface: "Every effort has been made to present the material in the simplest manner possible and the examples have been selected for their value in *illustrating the various techniques* rather than for their sensational value".

Professional men in the police departments and lawyers and judges will find much of real value in this book, while photographers, both professional and amateur, will realize that their fellow-photographers, who do specialized work in police laboratories or in forensic science laboratories, rank among the best exponents of photographic technique and ingenuity.

S. LAKSHMINARASU



# Central Advisory Council of Industries

**A**DDRESSING the first session of the Advisory Council (Jan. 24, 1949) the Hon'ble Dr. Syama Prasad Mookerjee, Minister for Industry and Supply, reviewed the industrial and economic problems of the country at the present juncture and the measures taken by the Government in this context.

The Government have taken preliminary steps for setting up or investigating into the possibility of establishing a number of important industries. The schemes include: the establishment of new steel works for increasing indigenous steel production by 1 million tons, construction of one or more ship-building yards, manufacture of synthetic petrol from coal, of heavy electrical equipment, of dry core, paper-insulated telephone cables, radio equipment, machine tools, diesel engines, tractors, penicillin, paludrine, sulpha drugs and explosives.

For increasing agricultural production, the Ministry of Agriculture has a number of schemes, the most important of which is the reclamation of waste land by mechanized cultivation. An area of 150,000 acres has already been brought under cultivation. The multi-purpose projects of the Ministry of Works, Mines and Power, when completed, will add over 27 million acres of irrigation to India's existing 48 million, and about 9 million kW. of hydro-electric energy to the present half a million.

Plans to develop rail and road transport, telephone and telegraph and schemes for port development and civil aviation are being executed in some cases and others are under investigation. The State-owned telephone factory, recently established at Bangalore, is designed to make India self-sufficient in regard to her requirements of automatic

telephone equipment and the factory near Asansol for the manufacture of locomotives is expected to be completed by the end of 1950.

The policy of the Government for raising the living standards of people is to encourage fruitful enterprises bringing quick results. To encourage cottage and small-scale industries, an All-India Cottage Industries Board has been set up and an all-India policy laid down by the Board is being implemented.

The Hon'ble Minister referred to the work of the Indian Standards Institution in 3 major fields of industry—cotton textiles, engineering and chemicals. He referred also to lack of statistical data and the measures the Government propose to take for the preparation of statutory annual census of manufacturing industries and compilation of monthly statistics.

As regards the nationalization of industries, the Government have assumed primary responsibility for the development of new undertakings in 6 industries other than arms and ammunition, railways and the production and control of atomic energy, viz. coal, iron and steel, air-craft manufacture, ship-building, manufacture of telephones, telegraph and wireless apparatus and mineral oils. The policy of the Government is specific in that the existing undertakings will not only remain with private enterprise for at least 10 years, but that they will be assisted to increase their efficiency and expand their production. The question of their acquisition by the State will be a matter for review at the end of 10 years in the light of the circumstances then prevailing.

The appended table gives the statistics of production for certain important industries.

## PRODUCTION TRENDS DURING 1947-48

*Note*—The 9-hr. day was generally in force till August 1945 as against the 8-hr. day now obtaining

SER. NO.	INDUSTRY	INSTALLED CAPACITY	PEAK PRODUCTION IN BEST YEAR	PRODUCTION IN 1947	PRODUCTION IN 1948	REMARKS
	Coal	30 million tons	30 million tons (1947)	30 million tons	29.73 million tons	
	Steel	1,264,000 tons	1,166,200 tons (1943)	893,296 tons	854,000 tons	Decrease in production due to labour troubles, transport difficulties and a temporary breakdown at Steel Corp. of Bengal (from April to Sept.)
	Salt	48 million md. (approx.)		49.6 million md.	50.3 million md.	Increase is due to the 10-acre concession for small-scale producers and favourable weather conditions
4.	Electric lamps	14.35 millions	9.13 millions (1948)	7.62 millions	9.13 millions	Increase due to new units
5.	Dry cells	142 millions	122.73 millions (1948)	87.91 million	122.73 millions	Increase due to new units
6.	Motorcar batteries	268,000	110,000 (1948)	70,028	110,000	Increase due to new units
7.	Electric motors	150,000 h.p.	60,000 h.p. (1948)	38,000 h.p.	60,000 h.p.	Increase due to new units
8.	Transformers	175,000 kVA.	80,000 kVA. (1948)	23,000 kVA.	80,000 kVA.	Increase due to new units
9.	Electric fans	250,000	180,000 (1948)	160,000	180,000	Increase due to new units
10.	Cables and wires	Wires (bare and covered) 24,350 tons Cable and Flexibles 50 million yd.	.....	.....	6,684 tons wire 23,913 million yd. (cables, etc.)	
11.	Electric black copper	24,000 tons	6944.1 tons (1946)	6,889 tons	3,630 tons	Decrease due to off-take of rubber insulated cable and bare copper wire



## PRODUCTION TRENDS DURING 1947-48 (contd.)

SER. No.	INDUSTRY	INSTALLED CAPACITY	PEAK PRODUCTION IN BEST YEAR	PRODUCTION IN 1947	PRODUCTION IN 1948	REMARKS
12.	Belting	1,600 tons	.....	615 tons	661 tons	
13.	Diesel engines	1,200	964 (1948)	761	964*	Increase due to new units
14.	Iron castings	400,000 tons	272,454 tons (1948)	168,333 tons	272,454 tons	Increase due to improved supplies of raw materials
15.	Machine tools	3,000 (1 graded)	2,900 (1945)	1,400	1,691*	Increase due to improved availability of raw materials and facilities for production
16.	Bicycles	60,000†	51,688 † (1948)	48,827	51,688†	Increase due to expanded capacity of existing units
17.	Sulphuric acid	100,000 tons	62,550 tons (1945)	60,000 tons	80,000 tons	Increase due to new plants
18.	Superphosphates	75,000 tons (1948)	21,358 tons (1948)	5,000 tons	21,358 tons	Low production in 1947 due to restricted supplies of imported rock phosphate which are now coming on a liberal scale
19.	Caustic soda	13,500 tons in 1948	4,388 tons (1948)	3,314 tons	4,383* tons	Increase due to certain units coming into production in 1948
20.	Soda ash	54,000 tons	28,200 tons (1948)	13,624 tons	28,200* tons	Increase due to improved methods of production and availability of raw materials
21.	Chlorine (liquid)	2,100 tons	1,825 tons (1945)	1,706 tons	1,800* tons	Increase due to improved off-take
22.	Bleaching powder	6,000 tons	2,836 tons (1948)	2,550 tons	2,836* tons	Production likely to increase further if greater supplies of good quality limestone are available
23.	Drugs (tinctures and galenicals)	750,000 gal.	600,000 gal. (1944)	500,000 gal.	522,000 gal.	Gap between capacity and production due to non-availability of the required quantity of spirit
24.	Metals					
	(i) Aluminium	5,000 tons	.....	3,215 tons	3,354* tons	Increase due to expansion in existing units
	(ii) Antimony	700 tons	.....	235 tons	370 tons	
	(iii) Copper	7,000 tons	.....	5,081 tons	6,031 tons	Normal fluctuation
	(iv) Lead	7,200 tons	.....	230 tons	545 tons	Industry not yet definitely established
	(v) Alloys	30,000 tons	.....	15,558 tons	15,732 tons	
	(vi) Semi-manufactures	50,000 tons	.....	22,545 tons	26,511 tons	Increase due to expansion in existing units.
25.	Rubber (raw)	17,174 tons (1944)	17,174 tons (1944)	16,449 tons	15,400 tons	Fall due to exhaustion of trees by slaughter tapping and stoppage of production on some estates due to unattractive prices
26.	Tyres and tubes					
	(i) For cycles	4.5 million sets	Tyres 3.34 millions (1948); tubes 4.32 millions (1947)	Tyres 3.23 millions; tubes 4.32 millions	Tyres 3.34 million; tubes 3.82 millions	
	(ii) Other than cycle (motor, giant, etc.)	1 million sets	Tyres 0.83 millions (1947); tubes 0.80 millions (1947)	Tyres 0.83 millions (1947); tubes 0.80 millions	Tyres 0.78 millions; tubes 0.74 millions	
27.	Alcohol					
	(i) Industrial	20 million gal.	.....	4.82 million gal.	4.9 million gal.	Increase due to additional plant
	(ii) Power	.....	.....	2.20 million gal.	3.54 million gal.	
28.	Raw cotton	Restrictions are in force over cultivation in view of food position		28 lakh bales	28* lakh bales	
29.	Cotton textiles					
	(i) Yarn	10,158,909 spindles (including 35,000 spindles installed during 1948)	1,642,831,000 lb. (1948)	1,814,524,000 lb.	1,442,342,000 lb.	Increase due to better use of capacity in mills during 1948
	(ii) Cloth	.....	4,735,750,000 yd. (1944)	3,816,088,000 yd.	4,337,502,000 yd.	
30.	Woollen manufactures (excluding cottage industries)	30 million lb.	30 million lb. (1945)	24 million lb.	23,668,074 lb.	
31.	Rayon weaving	150,000 hand looms	180 million yd. (1937)	85 million yd.	114 million yd.	Increase due to improved supplies of yarn
32.	Rayon weaving	15,000 power looms	88 million yd.	.....	.....	
33.	Cement	2,115,000 tons	.....	1,441,885 tons	1,516,226 tons*	Increase due to expansion of an existing unit
34.	Sugar	1,400,000 tons	1 million tons* (1948)	925,000 tons	1 million tons*	Increase due to higher price for sugarcane and sugar during 1948
35.	Paper and boards	110,000 tons	.....	93,090 tons	99,765 tons	
36.	Vanaspatti	220,000 tons	145,000 tons* (1948)	95,000 tons	145,000 tons	Increase due to new units
37.	Tea	551 million lb.	551 million lb. (1947)	551 million lb.	550 million lb.	Decrease due to climatic and labour conditions
38.	Coffee	15,040 tons (1947-48)	25,200 tons (1945-46)	15,040 tons (1947-48)	17,000 tons (1948-49; estimated)	Fall due to climatic conditions. Coffee has a 3 to 4 year cycle of good, medium, and poor crops

\* Approximate figures.

† Complete bicycles + spare parts equivalent to complete units.

PRODUCTION TRENDS DURING 1947-48 (contd.)						REMARKS
SER. No.	INDUSTRY	INSTALLED CAPACITY	PEAK PRODUCTION IN BEST YEAR	PRODUCTION IN 1947	PRODUCTION IN 1948	
39.	Cigarettes	30,000 millions	9,658.16 millions	18,879.77 millions	20,658.16 millions	Increase due to improvement in production and larger availability of raw materials
40.	Matches	800,000 cases (1 case = 50 gross boxes of 50 sticks each)	618,000 cases (1947)	618,000 cases	409,586 cases	For three-quarters only.
41.	Leather (production of organized units)					
	Buffalo hides (veg. tan)	2,000,000	.....	800,000	979,000	Decrease is due to shortage of tanning materials and transport
	Cow hides (chrome)	3,000,000	.....	1,200,000	1,087,000	
	Buffalo hides E.I. tanned	9,300,000	.....	6,420,000	4,602,300	
	Cow Hides Tanned	19,080,000	.....	17,500,000	15,250,000	
	Goat skins	63 million sq.ft. in 1948	50 million sq.ft. in 1944	28.6 million sq.ft.	38.63 million sq.ft.	Increase due to additional units
42.	Plywood	250,000 tons	190,000 tons (1948)	80,000 tons	190,000 tons	Marked improvement due to rapidly increasing availability of raw materials
43.	Soap	150,000 tons	120,000 tons (1945)	80,000 tons	70,000 tons	Decrease due to shortage of raw materials and fuel
44.	Glass	30,000 tons*	25,000 tons (1945)	20,000 tons	23,000 tons*	Increase due to additional plants
45.	Ceramics	225,000 tons (1948)	185,511 tons (1948)	175,376 tons	185,511 tons	Increase due to 3 firms going in for tunnel kilns
46.	Refractories	25 millions	.....	8.532 millions	6.763 millions	Fall despite increased capacity due to shortage of raw materials, particularly mild steel sheets and difficulties of transport
47.	Enamel ware	4,200,000	.....	.....	2,520,000	
48.	Insulators	121,680 reams	40,600 reams (1947 & 48)	40,600 reams	40,600 reams	
49.	Abrasives					

\* Approximate figures.

## Obituary—Prof. Birbal Sahni (1891-1949)

IN THE DEATH OF PROF. BIRBAL SAHNI ON April 9 at Lucknow, India has lost one of her eminent sons, and science a brilliant savant.

The 58-year old scientist was born at Bhera, Punjab, on 14th November 1891 and was educated at the Government College, Lahore and at the Emmanuel College, Cambridge. He was appointed Professor of Botany, University of Lucknow, in 1921, and this position he filled with conspicuous distinction all these years. He was the Founder and Honorary Director of the Institute of Palaeobotany, the foundation-stone of which was laid just a week before his death.

Among his numerous publications may be mentioned an excellent text-book of botany, original contributions on extinct plants, fossil flora and their geological bearings, and Indian numismatics. He was the Editor of the Lucknow University Studies, and Palaeobotany in India. He was a brilliant teacher esteemed alike by his numerous students, colleagues and friends both for his personal charm and scientific eminence.

He was elected a Fellow of the Royal Society, London, in 1936, President of the 27th Session of the Indian Science Congress, Madras (1940), and a non-official member of the Indian Delegation to the Royal Society Scientific Conference held in London in 1946. He was invited to preside over the Palaeobotany Section of the 7th International Botanical Congress to be held at Stockholm in July 1950. Prof. Sahni was awarded the Barclay Medal of the *Asiatic Society of Bengal* in 1936, and the Nelson Wright Medal of the *Numismatic Society of India*.

Prof. Sahni dedicated his private funds, his immovable property, a reference library and his valuable fossil collections for the promotion of research in fossil botany at the Institute of Palaeobotany at Lucknow. The cutting short of a fruitful career, at a time when the prospect of further activity on a large scale was opening out, will be deeply felt by all those interested in the progress of science in India.

# NOTES & NEWS

## A New Horizontal Electron Microscope

A HORIZONTAL ELECTRON MICROSCOPE with several distinctive features has been designed in the University College of Science, Calcutta (*Ind. J. Phy.*, 1948, **22**, 497). The instrument is essentially a research unit, flexible in design and highly suited for investigations on electro-optical problems. The different elements of the unit are mounted on 2 stainless steel rods held in position by brass sleeveings which can slide over the rods. It is thus possible to dismantle any part of the microscope without disturbing the rest. The distance between any 2 elements can be varied, and it is also possible to interpose an extra element between any 2 of the existing ones. Each microscope element is approachable from all sides, and the image formed on the fluorescent screen can be demonstrated to a number of people simultaneously. The instrument is designed for a maximum electronic magnification of 20,000 dia.

The electron gun, the condenser, the objective and the projector lenses are supported on separate carriages consisting of 4 pairs of horizontal brass plates. Any of the pairs of brass plates can slide together in a horizontal plane perpendicular to the axis of the microscope on a pair of stainless steel guide rods fixed to the frame of the instrument. The upper plates supporting the microscope elements can also be raised or lowered with respect to the lower plates by a set of screws. These 2 motions at right angles to the optical axis can be given to any element of microscope. By means of transmission gear arrangement, the operator, sitting at the control table near the final fluorescent screen, can move any of the lenses or the gun for proper alignment. Each of the 3 lenses has 4 levelling screws by means of which the lens may be slightly inclined to the axis so as to allow for any asymmetry of the pole pieces. In addition to lateral motions, the gun can be slightly tilted about the horizontal and vertical axes.

The length of the microscope column from the filament tip to the objective is 59 cm. and the length from the objective to the fluorescent screen is 79 cm. For evacuating the microscope column, an oil-diffusion pump and a *Cenco Hypervac 20* are used. The mechanical pump is housed in a specially designed underground chamber a little distance away from the microscope in order to reduce noise and vibration. A thermo-couple gauge measures the fore-vacuum while the high vacuum within the microscope is indicated by an ionization gauge.

The electron gun assembly is demountable and is made vacuum tight by means of the rubber gaskets and gasket tightening screws. For a change of filament, the filament unit together with the cathode shield can be taken out.

The object stage can be rotated through a small angle and the tilting of the stage for stereophotography purposes is also possible. 2 viewing ports provided on the upper side of the specimen chamber allow a view of the specimen stage through all operations. 4 hydraulic syphon

bellows, fitted at 90° to each other, provide movement for the object stage in 2 perpendicular directions at right angles to the optical axis. By compressing and expanding the hydraulic sylphons it is possible to move the stage in a plane perpendicular to the microscope axis and thus explore different parts of the specimen. It is also possible to move the stage while looking at the image on the final fluorescent screen. A unit is provided on the control desk with both coarse and fine adjustments, so that the specimen can be placed accurately in any desired position.

The specimen changing is accomplished by means of an air lock.

The photographic unit consists of a plate magazine holding 20 photographic plates and is demountable. The plates are moved by a pressure pad, the pressure being maintained by the vacuum in the unit. A mechanism is also provided by means of which the plate carrier and the shutter mechanism are swung out of the path of the electron beam and the total area of the fluorescent screen can be utilized for visual observation of the micrograph.

The microscope filament is heated by r.f. current of about 150 kc./s. This reduces the problem of electrostatic shielding and simplifies high voltage insulation.

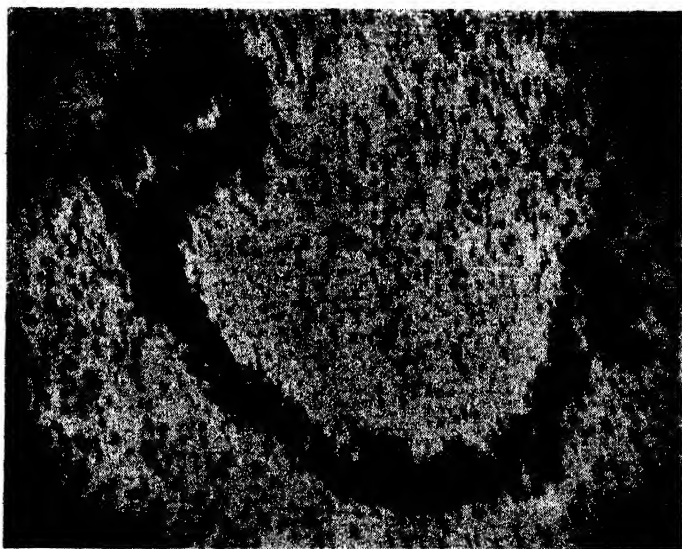


FIG. 1 — *LEPOSPIRA ICTEROHAEMORRHAGIAE*  $\times 10,000$ .

### Mounting Medium for Textile Fibres

AN IMPROVED PERMANENT MOUNTING medium for textile fibres is described (*Textile Res. J.*, 1948, 18, 756). A colourless permanent mount with the desired refractive index can be made by the combination of a synthetic resin of a moderately low refractive index with a compatible plasticizer of a much higher index. By varying the composition of the mixture, it is possible to adjust the refractive index to a value suitable for a particular fibre. A mounting medium with a refractive index that is approximately half-way between the maximum and minimum indices of common fibres has been found to be most useful.

Isobutyl methacrylate polymer (*Du Pont*) of refractive index 1.48 and Aroclor 1242 (*Monsanto*) of refractive index 1.62 are, respectively, the resin and the plasticizer used for the mount medium. Both materials are colourless and quite miscible with xylene. The proportions of all the 3 materials are adjusted until a viscous liquid with a refractive index 1.52 is obtained. This index is half-way between the indices of cotton or wool and acetate rayon.

The following is a formula for a permanent mounting medium (after heating) with a refractive index of 1.516 and 1.520:

Xylene (filtered)	21 c.c.
Aroclor 1242	9 c.c.
Isobutyl methacrylate polymer	18 gm.

The isobutyl methacrylate polymer should be finely ground and free from lumps. Into the filtered xylene Aroclor 1242 is first added and then the methacrylate polymer and the contents stored. The mix is allowed to stand overnight until all the bubbles produced due to stirring have passed off. The resulting colourless liquid has a viscosity very close to that which is usual with solutions of Canada balsam. The material is used in the same manner as Canada balsam.

A slight excess of material is employed and the slide after preparation should be heated in an oven at 70°C. for at least 2 hr. The excess material around the cover glass can be removed by a razor blade.

The 2 tables below give the approximate refractive indices of various mixtures, and data for obtaining photomicrographs of fibres using the mixtures.

### Manufacture of Anhydrous Hydrofluoric Acid

THE CONSTRUCTION AND OPERATION of the first plant to produce anhydrous hydrofluoric acid by a continuous process at the *Imperial Smelting Corp.*, Avonmouth, England, are described as a landmark in chemical history (*Ind. Chem.*, 1948, 24, 801). The anhydrous acid is for the first time transported in rail-tank cars. The plant is producing 6 tons of 80 per cent acid per day.

The major problem in the manufacture of anhydrous

hydrofluoric acid is corrosion, and the process is, therefore, of particular interest from the point of view of materials of construction employed. At strengths of 80 per cent or over of the acid, little difficulty is experienced from corrosive attack so that the section of the plant in which acid at these high concentrations are handled can be constructed of mild steel. Below 80 per cent corrosion is very severe and it has been necessary to employ plastics in various forms of Karbate, a form of non-porous carbon.

Fluorspar, a flotation product of acid grade and mostly passing — 200 mesh and analysing to 97.0 per cent  $\text{CaF}_2$  and less than 1 per cent  $\text{SiO}_2$  and  $\text{CaCO}_3$  and concentrated sulphuric acid are heated in a reaction kiln at about 430°C. and the gas evolved is first absorbed in 80 per cent acid and subsequently in 60 and 30 per cent acid. The reaction kiln is a cylindrical mild-steel structure (6' dia.  $\times$  40' long), supported on 2 steel tyres running on rollers and rotated at about 1 r.p.m. All extraneous air is sealed off from the kiln by means of pressure oil seals.

The gas stream is washed in counter-current with sulphuric acid in the "drip acid tower" constructed of mild steel and then absorbed in 3 stages as described above. The absorption towers are of mild steel and of the disc and plate type and are cork-lagged. The circulating acid is cooled by passing it through brine coolers of mild steel in the first stage of absorption. The circulating acid is diluted in a bronze pot. The second absorption tower containing 60 per cent acid comprises a Keebush tower, a circulating pump of Karbate and water cooler constructed of Karbate pipes. Keebush has been selected as the material for the tower, as it is capable of withstanding 70 to 75 per cent acid satisfactorily. The 30 per cent acid tower is similar to the 60 per cent one but is made of Plastimold.

Anhydrous acid is obtained by distilling 80 per cent acid in a simple bubble-cap type distillation column of copper. The vapour stream is cooled in a mild-steel brine cooler and stored in mild-steel tanks.

The anhydrous acid is delivered directly to rail cars (6.5 tons capacity) and is effected by means of compressed air.

TABLE I — APPROXIMATE REFRACTIVE INDICES OF VARIOUS MIXTURES

ISOBUTYL METHACRYLATE POLYMER gm.	AROCLOR 1242 c.c.	XYLENE c.c.	REF. INDEX OF FINISHED MOUNT
18	...	30	Between 1.495 and 1.500
18	3	27	" 1.495 and 1.500
18	6	24	" 1.505 and 1.510
18	9	21	" 1.515 and 1.520
18	12	18	" 1.520 and 1.525

TABLE II — DATA FOR PHOTOMICROGRAPHS OF FIBRES

FIBRE	REF. INDEX	MOUNTING MEDIUM	REF. INDEX	VISIBILITY
Cotton	1.56	Canada balsam	1.54	Poor
Cotton	1.56	Formula with 9 c.c. Aroclor 1242	1.515-1.520	Good
Cotton	1.56	Formula with 3 c.c. Aroclor 1242	1.495-1.500	Good
Acetate rayon	1.48	Formula with 3 c.c. Aroclor 1242	1.495-1.500	Very poor
Wool	1.55	Canada balsam	1.54	Poor
Wool	1.55	Formula with 9 c.c. Aroclor 1242	1.515-1.520	Good
Wool	1.55	Formula with 3 c.c. Aroclor 1242	1.495-1.500	Good

For pipe lines Saran has been employed and in some cases polythene has shown considerable promise. 60 and 30 per cent acids are stored in tanks lined with Neoprene. For the concentrated acid, valves with phosphor bronze bodies and Ilium seats have proved adequate. A satisfactory pump-packing material has been the only difficulty so far.

### Measurement of Radioactive Isotopes

THE DETERMINATION OF THE NUMBER of radioactive atoms in a sample of a radioactive isotopes has become a matter of importance, because of their expanding use in biological and medical studies, and in the clinical treatment of diseases. Uniform quantitative results can be obtained only by using procedures that will give the same result whenever the determination is made. The *National Bureau of Standards*, U.S.A., has, therefore, issued standard samples of radio-isotopes whose radioactivity has been accurately determined and is planning to issue samples of additional radio-isotopes as they become available (*J. Franklin Inst.*, 1948, 246, 419).

Two factors involved in the preparation and use of the samples are: (i) methods and standards that are correct in principle must be used; and (ii) a reasonable degree of accuracy must be attained in all steps of the process both of producing and of using the standards.

Most of the difficulties in making quantitative determinations of radio-isotopes can be eliminated if standard sources of the isotope are available. Such a standard source consists of a preparation of the isotope in a form convenient for use with the detector of radiation and for which the disintegration rate measured is the same as that from which the standard is made. Reliable results may be secured by observing only a few simple precautions. Isotopes which have short half-life periods and a known disintegration scheme may be measured with a fair accuracy by means of  $\gamma$ -ray standards, if it is known that a  $\gamma$ -ray or a positron is emitted for every disintegration, and if the maximum energies of  $\gamma$ -ray specific of the standard and of the specimen are not too widely different. In the case of those isotopes for which a

disintegration scheme is lacking, or for which the mode of disintegration does not permit measurement of disintegration rates, an alternative method of comparing the activities of sources is available if the isotope emits  $\gamma$ -rays. This method is, however, limited to comparisons of the same isotope. An ionization instrument properly designed to measure Roentengen units will satisfy the requirements for a standard instrument. To determine the strength of a radioactive source the Roentengens per unit time must be measured at a standard distance. On the basis of these considerations, the *Bureau* scientists have proposed that the unit of time be 1 hr. and the distance 1 m., giving as a unit for comparison of  $\gamma$ -ray sources the *Roentengen per hour at a meter (r.h.m.)*.

The name *Rutherford (rd)* has been proposed for that quantity of radio-isotope which disintegrates at the rate of a million disintegrations per sec. This unit which is smaller than the *Curie* can be specified exactly and independently of natural constants such as the rate of decay of radium, which is the basis of the *Curie*. The *Bureau* has adopted an arbitrary value of  $3.700 \times 10^{10}$  disintegrations per sec. for the *Curie* when applied to isotopes other than the members of the radium family. The use of *Rutherford* in data presupposes that a disintegration rate has been measured and that this rate is expressed in disintegrations per sec. This procedure removes all confusion regarding units and renders data from several laboratories comparable on an absolute basis.

### Radio-cardiography

A NEW METHOD FOR STUDYING blood flow through the chambers of the heart has been developed by recording graphically the passage of radioactive substances through the cardiac chambers with the aid of a specially constructed, ink-writing Geiger-Muller counter (*Science*, 1948, 108, 340). The procedure makes possible the investigation in human beings of certain haemodynamic functions not previously accessible to study.

The method consists in placing a carefully shielded Geiger-Muller counter over the pericardium, rapidly injecting 0.1 to 0.2 mc. of radio-sodium ( $\text{Na}^{24}$ ) into one of the antecubital veins, and

recording the counts by means of the newly devised direct-writing counter. Of the 2 waves recorded on the radio-cardiogram, the first wave relates to the entrance of the blood into superior vena cava, right auricle and the right ventricle and the pulmonary circulation and the second wave represents the period during which the left cardiac chambers are being emptied of the labelled blood.

The tracings taken in subjects with enlarged hearts and failure do not appear to differ materially from those in cardiac enlargement without failure. This observation arouses interesting speculation concerning the role of the heart in failure. Other observations of interest emerging from these studies are: the rate of venous return from a lower limb is much slower than that from an upper extremity, which helps to explain the greater tendency of venous thrombosis to develop in the lower extremities. The recording counter has also been used to estimate the time required for a substance to be absorbed from the side of a local infection; in preliminary studies on congenital heart disease; and in revealing certain sources of error in the usual clinical methods of determining circulation time and provides a more accurate method of determination.

### Improved Seismic Method of Locating Oil

AN IMPROVED METHOD OF locating underground petroleum deposits has been reported by Dr. Thomas C. Poulter of the *Institute of Inventive Research*, San Antonio, Texas. The method employs "shaped charges" of explosive compositions which are set off above the ground, thus eliminating the cost of drilling shot holes. The "shaped charge" is based on the principle that if a hollow is made in an explosive cartridge on the side toward the object to be blasted, the effect is greatly increased. The method can be used in regions where boring would be exceedingly difficult as in mountainous country or in a search for oil under sea.

Charges are set close to the ground and spread in a hexagonal design. Depending upon the type of record sought, comparatively light charges are placed from 5' to 85' apart and detonated simultaneously. The procedure may be used to produce an

essentially flat wave front of low amplitude over a relatively large area (*Science Newsletter*, 1948, Nov. 13, p. 307).

### Growth Factor in Coconut Milk

THE STRIKING NUTRITIONAL effect of coconut milk on the growth of explants of carrot root has been demonstrated both in liquid media and on nutrient agar (*Science*, 1948, **108**, 655). This nutritive fluid endosperm has already found application in the culture of bacteria, fungi, orchids and immature plant embryos. It has been claimed by a number of workers that it contains both a specific factor for plant embryo development and a thermo-stable general growth factor, probably indole-3-acetic acid. The evidence, accumulated so far, favours the presence of an unidentified active principle in coconut milk which can foster rapid growth of carrot cultures. Qualities which promote growth of carrot cultures can be demonstrated in heat-sterilized, filtered, water-clear preparations of coconut milk ( $pH$ , 5.6-5.9) obtained from mature nuts. Other experiments have shown that 1 per cent by volume of coconut milk added to an otherwise complete organic and inorganic nutrient medium causes a marked increase in growth; at about 15-20 per cent there is an optimum with marked decrease at higher concentrations. No growth occurs in coconut milk alone. In the presence of indole acetic acid alone a mean increase in weight observed was 8.2 mg. in 21 days and with added coconut milk a mean weight of 184 mg. was observed.

The active principle of coconut milk is not a constituent of the ash; nor is it any of the other known growth factors: thiamin, niacin, pyridoxine and glycine. It seems also to be different from any of the normal constituents of yeast, malt, tomato, carrot or liver extract when used at  $pH$  5.6. Though coconut milk may contain indole acetic acid, it is clear that this is not the factor responsible for increased growth. The substance is stable to prolonged autoclaving and, since its activity decreases on dialysis, it has relatively small molecules.

The work done so far points to much wider problems than those which merely affect growth,

and the following points suggest themselves:

(i) An outstanding difference between the carrot cultures which do not grow in the absence of coconut milk, or which sluggishly expand in the presence of indole acetic acid, and those which burst into very rapid growth must surely be in their ability to harness their respiratory energy in protein synthesis, salt and water uptake, i.e. to maintain the process in the cells which require the use of energy. It would be reasonable, therefore, to seek this coupling mechanism in some effect of the coconut milk which may promote not only carbohydrate breakdown, but also the linkage of respiratory energy to useful work; (ii) the possibility exists, however, that the active principle in coconut milk is not peculiar to carrot tissue and is at least specific for cells which contain carotenoids.

The literature on vitamin A also suggests a possible connection between a constituent of the coconut milk and vitamin A or carotene. Apparently coconut milk, as a partial source of protein in the basal vitamin A deficient diet, causes the vitamin A to be more effectively used when it is supplied: It has been suggested that there is a factor in coconut milk which, combined with vitamin A, makes the vitamin more effective or which facilitates the transformation of carotene to vitamin A.

### Geochemical Prospecting

AMONG THE NEWER METHODS OF investigation to discover mineral deposits is the geochemical method which is being increasingly employed in Australia (*Chem. Age*, 1949, **60**, 163). Australia is particularly suited to such methods of investigation because the mature residual soils of parts of the country and possibly some non-residual soils are likely to contain geochemical anomalies in mineralized areas, such anomalies being related spatially and genetically to economically important ores. It is likely that these methods will be most useful in areas where neither geological nor geophysical methods can indicate ore bodies.

Most of the claims in this field have come from Russia where deposits of tungsten, molybdenum and tin were claimed to have been discovered by

examining spectrographically soils up to 9' thick. A geochemical unit in U.S.A. is engaged in field, laboratory and greenhouse research, chiefly on copper and lead contents of soils, plants and waters in areas of known mineralization.

The poorly "dispersed" elements such as tin, lead, mercury, etc., may produce well-defined dispersion halos at no great distance from their source, both in semi-arid and in humid environments. The "dispersed" elements such as zinc, nickel and others may form large but poorly defined halos; in areas of ample rainfall these "dispersed" elements may travel long distances.

Four methods are employed: (1) the soil samples are tested by colour reactions to indicate abnormal concentrations; (2) spectrographic analysis of soils, which has proved of limited value; (3) the role of plants as guides to prospecting which takes into account the fact that certain plants grow best when there is a high concentration of a metal, and the accumulation by plants in their tissues of metals such as zinc, nickel, copper and cobalt in the leaves and lead and arsenic in roots; the available experience in U.S.A. indicates that plants, in general, are not precise indicators of geochemical anomalies; (4) testing of surface and ground waters in order to locate unusually high concentrations of heavy metals. The examination of water as a procedure of general exploration in a virgin country may be a great time-saver.

### A Rapid Laboratory Method for DDT

DDT, AS PREPARED BY THE Bayer condensation method using chloral and fuming sulphuric acid, is expensive and requires complicated apparatus. Both chloral and fuming sulphuric acid are unpleasant and hazardous to handle, especially by inexperienced workers and students. A short and simple method for preparing small quantities of DDT without the use of either chloral or fuming sulphuric acid has been worked out to overcome the above drawbacks (*Science*, 1948, **108**, 339). The entire process can be carried out within one laboratory period and has been used by graduate students taking courses in insecticides.

The method is based on the theoretical reaction of 1 mole

of chloral hydrate and 2 moles of chlorobenzene (sp. gr., 1.107) in the presence of about 4 to 5 times their combined volumes of concentrated sulphuric acid.

17 gm. of chloral hydrate crystals and 23 c.c. of chlorobenzene are placed in a glass-stoppered, 500 c.c. pyrex reagent flask and kept in an electric oven at 60°-70°C. for about 20-30 min. with occasional shaking or until all the crystals have dissolved. The contents are cooled to room temperature and about 180 c.c. of concentrated sulphuric acid added, the flask stoppered and contents vigorously shaken until precipitation starts. This operation requires about 1 hr. The contents are let stand for 15 min. with an occasional shaking or until precipitation is complete. The mixture is poured into a glass jar containing about 4 litres of cold tap water and allowed to stand for 15 min. or until the solids have settled down. The precipitate is filtered through 3 layers of cheese cloth, washed several times with water and transferred to a glass jar or wide-mouthed bottle to which 50 c.c. of either 2 per cent sodium carbonate or 4 per cent sodium bicarbonate solution are added to neutralize the acidity. The product is finally filtered over a Buchner and washed with distilled water until the filtrate is neutral to litmus. The residue is triturated with 100 c.c. of ethanol in a mortar and pestle for about 10 min. and filtered in a Buchner and dried in an electric oven at 70°-75°C. for about 2 hr. or until all the alcohol has volatilized. An average yield of about 16 gm. of pure DDT is obtained and analyse to an average of 96.82 per cent *p,p'* DDT with an m.p. ranging from 106°-108°C.

### Fluorine Substituted DDT

ANOTHER INSECTICIDE, DIFLUORODIPHENYL-TRICHLOROETHANE (DFDT), a compound obtained by substituting 2 chlorine atoms in DDT by fluorine, has proved to be better than DDT in killing house flies and certain other insects. Extensively employed in Germany during the war, the new insecticide is less toxic to warm-blooded animals and fish. Although it kills fewer insect species than DDT, it has quicker knock-down power against those it is effective (*Sci. Newsletter*, Jan. 1, 1949, p. 9).

### Estimation of Rotenone

A RAPID AND SIMPLE METHOD for evaluating the toxicity of *derris* roots is based on the absorption of light by the toxic constituents of the roots dissolved in acetone and measuring it in a spectrophotometer at 360 m $\mu$  (*J. Agr. Res.*, 1948, 77, 271).

The method is as follows: 1 gm. of *derris* root, ground to pass an 80-mesh sieve, is taken in a 125 c.c. Erlenmeyer flask with 50 c.c. of purified acetone. The contents are swirled for a few seconds and set aside for 1 hr. or longer with occasional shaking. The contents are cooled in an ice-box for 30 min. and then rapidly filtered taking every precaution to reduce the evaporation of the solvent. When the filtrate reaches room temperature, a 10 c.c. aliquot is made up to 100 c.c. in a volumetric flask using pure acetone. The percentage of transmittance at 360 m $\mu$  is measured in a spectrophotometer using pure acetone as reference solution. The rotenone equivalent is determined from a standard curve prepared from transmittance values of 2 biologically assayed *derris* roots prepared as above.

When acetone extracts of *derris* samples are adjusted to equal rotenone contents and the percentage transmittance determined, there is close agreement between the known content of other toxic constituents (rotenoids) and the transmittance values. Likewise, transmittance values of graded rotenone solution in acetone determined at 360 m $\mu$  follows the Beer's-Lambert law. The transmittance values of total acetone extracts of *derris* roots are in close agreement with the rotenone equivalents as determined biologically on house flies. Transmittance values of *Lonchocarpus* root do not correlate with the biological rotenone equivalent indicating that some of the toxic constituents in these roots are different from those in *derris* roots.

### Humidity Test Apparatus

A "DIVIDED FLOW, LOW TEMPERATURE test apparatus", developed at the National Bureau of Standards, now makes available an instrument for research, calibration and testing of hygrometers at temperatures below freezing (*J. Franklin Inst.*, 1948, 246, 421). The use in recent years of radiosondes for the meteorological

sounding of the atmosphere has created a marked need for information on the performance of the humidity-sensing element in the radiosonde under conditions of flight.

The function of the humidity test apparatus is to produce air of known relative humidity at temperatures from 0° to -40°C. In this instrument, a current of dry air is divided into 2 streams, one of which is maintained dry while the other is saturated with respect to ice; finally the two are recombined. A proportioning valve divides the flow of moisture-free air in a known ratio. One part passes through a saturator, containing ice-trays till saturated, then mixed in a mixing chamber with the other dry part and finally allowed to exhaust through a test chamber into the atmosphere. The saturator, mixing chamber and the test chamber are kept immersed in a constant temperature bath. The hygrometer to be tested is inserted into the test chamber for testing. The overall performance of this apparatus has been established by a series of gravimetric moisture determinations. For the temperature range of 0° to -40°C. and for air velocities up to 1500' per min., this instrument produces air whose relative humidity is known within 3 per cent.

While particularly suited for investigation and calibration of hygrometers, psychrometers and development apparatus at temperatures below 0°C., this equipment may find further application in general research and testing where air of a known low dew-point or moisture content is desired. With such apparatus, devices placed in the test chamber can be subjected to discrete and almost instantaneous changes in relative humidity. It may also find useful application in refrigeration and air-conditioning industries.

### Flax Retting with Aeration

THE NORMAL FLAX RETTING process, which is anaerobic, gives rise to a large volume of polluting waste waters which present a serious disposal problem. In a factory with a capacity of 32 tons of flax straw per week, an average of 40,000 gal. a day of waste waters are discharged from the tanks which is equivalent to about 170,000 gal. of settled domestic sewage. Further, the disposal of waste waters from ret-



ting present far greater difficulty than would be encountered in the disposal of an equivalent volume of domestic sewage because of their acid nature and the polluting matter is in colloidal dispersion and cannot be removed by simple sedimentation. *Technical Paper No. 10*, published by the *Water Pollution Research Laboratory, D.S.I.R.*, London (*H.M.S.O.*, price 3s.), gives an account of the investigation, of which the final stage was the development of a new process in which the retting liquor is aerated during retting and re-used.

The process may find application in the retting of other fibrous materials like jute, coir, etc.

In laboratory-scale experiments, it was shown that the waste waters from anaerobic retting treatment could be treated economically to give an effluent of a high quality. In small-scale experiments good results were obtained when successive batches of flax were retted in the same liquor which was treated continuously by passage through percolating filters or through a separate aeration tank or which was aerated in the retting tank itself during retting. The fibre produced by all the 3 methods was of good quality; the method of using biological filtration produced a fibre of a better quality than that from other processes, but the area of filters required would be large. For a rettery with 4 tanks, each holding 4 tons of deseeded flax, an area of about 4,050 sq. ft. of filters, 6' deep, would be necessary.

During all the stages of the work, bacteriological examinations were made of the retting liquor and of the flax during retting, and much information on the bacteria responsible for retting was obtained. It was found that, when the liquor was aerated, bacterial growth resulted largely in the organic matter being oxidized away instead of being converted to acids as in the anaerobic process. Conditions in the flax stems themselves, however, were still anaerobic and the organisms mainly responsible for retting, even under conditions of considerable aeration, were found to be spore-forming anaerobes.

A number of flax factories in England are operating plants employing retting with aeration. The tanks usually are 4 in number, of 4 tons capacity of

deseeded flax each, rectangular and, in its internal dimensions, 19' 8" long, 13' wide and 9' 3" deep. The top is open and is fitted with transverse cross beams 8" apart to retain flax. On the floor are diffuse units through which air is bubbled through the retting liquor, and above are wooden grids on which flax is packed. Equipment for injecting steam to heat the liquid in the tank is provided.

A storage tank is provided, a third more in capacity than that of the retting tank, into which liquor from the retting tanks can be run in. The heating here is by steam injection.

Compressed air is supplied to the tanks by 2 compressors, each driven by a 6 h.p. electric motor and rated to deliver 100 cu. ft. of air per min. against a pressure of 4.5 lb. per sq. in. Each diffuser unit has a separate air pipe and the distribution of air can be adjusted by a valve on each pipe.

Before the start of a ret, water is run into the tank to a depth of about 6" above the diffuser plates, and the air supply is turned on and adjusted to give even distribution. Beets of deseeded flax are packed into the tank in 2 tiers. Fresh water, which has been heated in the storage tank to 30°C., is run into the retting tank to within about 3" of the top and the air supply is increased during the filling to about 30 cu. ft. per min. The temperature of the liquor in the tank is maintained at about 30°C. and warm fresh water or retting liquor is added during the ret to maintain the level of liquid in the tank.

Retting is generally complete in 2 to 3 days at 30°C. Samples are withdrawn at frequent intervals after the first 24 hr. for examination; when the fibre is found to be separating from the straw, samples should be withdrawn at least every hour for examination.

When a ret is finished, the liquor is run off into the storage tank, leaving enough in the retting tank to cover the diffuser plates and retted flax removed. The retting tank is then packed with fresh straw, the liquor is pumped back from the storage and fresh water is added to make up the original amount.

Samples of retting liquor are analysed 3 or 4 times a day for the acidity or alkalinity. If acidity increases, the air supply

is increased; if the liquor becomes alkaline, the air supply is reduced.

Aeration should be maintained between rets so long as the diffuser plates are covered with retting liquor. At least once a week all liquor should be run off and the wooden grids removed so that material deposited from the flax can be forked and hosed out.

The bulletin has a useful chapter on review of literature on retting of flax and on the various aspects of retting and methods employed in several countries. The 3 appendices relate to bacteriological investigations and methods of analysis.

### Continuous Steel-casting

ONE OF THE FAR-REACHING developments in steel-casting, which promises to revolutionize the steel industry, is the continuous steel-casting method developed by Prof. Sachs, Director, National Metallurgical Laboratory, prior to his coming to India. The new method dispenses away with the reheating and rolling process of the conventional process and saves space and expense.

In this process the steel is poured into an oval-shaped mould, where it starts to solidify uniformly and quickly. The steel flows from the mould through a sleeve-like insulated chamber below the mould, which equalizes the temperature of the steel as it further hardens. As the metal is withdrawn, it is cut into billet-size length by an automatic oxy-acetylene torch. The steel, then in a semi-finished shape, is ready to be sent to the finishing mill.

One of the most difficult tasks in the development of the process was finding the correct oval shape for the mould. By the use of zinc and aluminium alloys the behaviour of steel in the oval mould was modified and the correct mould shape facilitating the freezing of steel was determined.

### Improved Baker's Yeast

A NEW PROCESS FOR THE MANUFACTURE of baker's yeast, the Deloffre Alcohol Process, named after the inventor, promises to improve world's yeast industry, according to a recent Australian report. The yeast, in this process, is made in one operation by inoculating a highly concentrated solution with a culture of



pure yeast, which is then fermented to set up the required biological reactions under which yeast can absorb alcohol.

The advantages of the process are many. Due to the high alcohol concentration in the fermenting liquid, fermentation takes place under sterile conditions without any bacterial contamination. The manufacturing process is simplified, the production cost cut by 30 per cent, yield increased by 20 to 25 per cent, and the yeast produced has exceedingly high keeping qualities because high alcohol concentration reduces the protease content of yeast, and has better baking properties with substantially greater leavening power.

The entire process is automatic and a special feature of the plant is that the pipe lines conveying the materials are kept to a minimum length.

Australia uses about 8,000,000 lb. of yeast each year for baking purposes and 80 per cent of it is produced by the Deloffre Alcohol Process (*Agricultural Newsletter, Australia*).

#### New Varieties of Fish in Rihand River

AN UNKNOWN SPECIES OF FISH and several other specimens of exceptional zoological interest have been discovered in a fish survey of the Rihand river in the Mirzapur district (U.P.) recently carried out by the *Zoological Survey of India*. 42 varieties of fish were found in the neighbourhood of the site of Pipri dam. 16 of these are of economic importance, though all of them are edible. The South Indian *Khudree Mahseer* found in the river grows to a large size and may be found suitable for large-scale cultivation in the dam reservoir. The temperature is also likely to suit the culture of Indian carp. It is estimated that if scientifically stocked and judiciously exploited, the reservoir may yield over 1,300,000 lb. of fish annually.

#### Wind Power for Agriculture

THE *Monthly Weather Review* published by the Government of India shows average and normal wind velocity at 8 a.m. and 5 p.m. at hundreds of weather stations throughout the country. Unfortunately these hours are unsatisfactory for measuring windmill possibilities, because wind velocity is then much less than

during the other hours of the day. At Belgaum (Bombay Province), for which hourly data are available, the monthly averages are 2 to 3 times as great as the average velocity at 8 a.m. and 5 p.m. recorded in the Review. Nevertheless, these data give a rough indication of the places where more wind is obtained and months when winds are strongest. Possibly part of the seasonal variation is due to the fact that wind strengths are greater at an earlier hour and dies down at a later hour when days are longer.

The normal wind velocities recorded at 3 stations in India (TABLE I) give a fair idea of the wind velocities.

The records at other stations show that India has enough wind power to operate certain types of American windmills almost everywhere except in the Gangetic plains. The best places for windmills are in the draught-stricken Saurashtra and almost anywhere between Karachi and southern Bombay province. To this should be added parts of coastal region along the Bay of Bengal and much of Deccan plateau and South India.

A pamphlet published by the Director General of Observatories, *Wind Data for Windmills*, reports experiments conducted in Madras

which shows that a steady breeze of about 6 miles an hour would keep a windmill in continuous operation pumping water from a depth of 25'. A 4-mile breeze at Coimbatore was shown to pump enough water for domestic purposes.

In more detailed basic data available for 12 points in India with wind velocity recorded every hour, several other favourable factors are revealed (TABLE II). Wind velocity is greater during day-time when it is convenient to irrigate the crops. There is generally more wind during the hot months preceding and at the beginning of the monsoon rains when it is most important to get the crops sown and growing, and there is scarcely a month without sufficient wind power not to justify a windmill except at Agra, Jubbulpore and Calcutta.

The data show that in a year a windmill pumping water for 2,000 hr. will be pumping water for more hours than a pair of bullocks do. A windmill of the American type described in the United States Department of Agriculture, *Farmer's Bulletin No. 1857* (December 1940), costs just a little more than a pair of bullocks and can easily be worked on wind velocities of 4 to 6 miles an hour. A windmill pumping

TABLE I.—NORMAL WIND VELOCITY: MILES PER HR.  
AT 8 A.M. & 5 P.M.

	RAJKOT		BANGALOR	
January	4.1	5.7	5.1	
February	4.8	5.5	4.7	
March	6.0	5.7	4.5	
April	8.1	6.4	4.3	
May	11.3	8.2	5.5	
June	11.7	8.2	8.2	
July	12.0	10.1	8.4	
August	10.2	8.7	7.3	
September	7.0	6.4	5.8	
October	4.2	5.0	4.1	
November	3.6	6.1	4.4	
December	3.6	5.7	4.8	

TABLE II.—WINDY DAYS & HOURS DURING A YEAR

(Average of 5 years)

STATION	DAYS WITH WIND BLOWING AT LEAST 6 MILES PER HR. FOR 10 HR. OR MORE	HR. OF WIND BLOWING AT LEAST 7 MILES PER HR.
Dhubri (Assam)	91	2,190
Sagar Island (Calcutta)	263	5,782
Calcutta (Alipore)	40	1,051
Allahabad	116	2,628
Agra	40	1,139
Deesa (Rajputana)	226	4,643
Jubbulpore	69	1,752
Bombay	223	4,468
Belgaum	307	6,132
Bangalore	248	5,081
Madras	182	3,767
Kodaikanal	212	4,380

500 gallons a day per hour for 13 hr. a day will deliver 2' of water to an acre of land within a week.

The factory price of an American 40' windmill tower in U.S.A. is about \$150 and the price of a wheel and accessories range from \$100 for the 6' size to about \$300 for a 12' size.

Too much should not be expected from wind power. If the required flow of water is over 400 cu. ft. per hr. or if the lift is much over 50', it is the electric power or fuel power that can do the job (*USIS*).

### Non-ferrous Metals Development Committee

THE COMMITTEE HELD ITS SECOND meeting during the last week of January and, after reviewing the general overall position of the non-ferrous metallurgical industries in the country, fixed the following target figures for the non-ferrous metals and alloys with the ultimate objective of attaining self-sufficiency in respect of semi-manufactured metals and alloys (TABLE below).

	6,000/7,000 tons per year
Copper (fire refined)	20,000 " "
Copper (electrolytic, from copper base scrap)	6,000 " "
Copper sheets	35,000 " "
Brass sheets	5,000 " "
Copper and brass tubes and shapes	6,000 " "
Lead	7,000 " "
Antimony (depending upon imported ore)	15,000 tons, short-term target
Aluminium ingots	50,000 tons, long term target
" sheets	10,000 tons, short-term target
" sheets	25,000 tons, long term target
" foil	1,500 tons per year
" powder	1,300 " "
" wire and cables	5,000 " "
" tubes and shapes	2,000 " "

As regards white metal and copper-base alloy ingots and castings, it was stated that there was already adequate capacity in the country to meet any demand including exports.

To attain the above target figures, the Committee recommended to the Government the need to take immediate steps to have a comprehensive project about the setting up of an electrolytic copper refinery to treat copper-bearing scrap, as at the present moment India had to depend entirely on imports for her requirements of electrolytic copper. This move is deemed essential not only on account of the strategic importance for the defence of the country but also for peacetime industrial development. The Committee also recommended

the need for Government assistance for the development of the Zawar lead-zinc mines, and for the procurement of antimony ore to the smelter at Bombay, which had now to depend entirely on imported ore. India has ample resources for increasing aluminium production and, therefore, the Committee emphasized the need to formulate a definite policy regarding this industry. But as any scheme of expansion badly needs finance, which at the present moment is difficult to raise, the Committee felt that it was most essential for the Government to create favourable conditions to encourage investment in industry.

### Committee on Factory Data Reporting

A SPECIAL COMMITTEE OF THE *International Society of Sugar-cane Technologists* on uniformity in reporting factory data has been formed with the following members: Dr. F. W. Zerbán (New York), Chairman, and Messrs Alejandro S. Alvarex (Argentina), Anibal R. de Matos, Hoao da

tation to promote effective exchange of research and development information among the departments of the National Military Establishment was announced recently by Dr. Vannevar Bush, Chairman of the Research and Development Board, National Military Establishment, U.S.A. The Committee will study the problem of collecting, correlating, reproducing and disseminating technical information potentially useful in the research and development programme of the National Military Establishment. Study and application of new methods and techniques to the problem of technical information organization and promotion of active research in this effort are to receive special attention by the group.

### Herbage & Fodder Plants

BULLETIN 39 OF THE *Australian Commonwealth Bureau of Pastures & Field Crops*, entitled *Five Hundred Varieties of Herbage and Fodder Plants* (edited by M. Hall; Price 18s. 9d.), is a descriptive catalogue of recognized species and varieties of fodder plants. Under each variety details are given of its origin, the authority responsible for its development, its characteristics, adaptation, use and certification, together with notes on the market for seed. Species are arranged alphabetically and varieties are grouped according to the country of origin or use. Australian species are covered better than that of other countries.

The Bulletin will be of particular interest to persons concerned with the introduction and breeding of pasture plants and forage crops, and will also be a useful document for agriculturists and seedsmen who give advice on the choice of varieties for specific purposes. Copies of the publication can be had from the Liaison Officer, Commonwealth Agricultural Bureaux, 314 Albert Street, East Melbourne, C. 2, Australia (*J.C.S.I.R.*, 1948, **21**, 223).

### Wool Research, 1918-48

*The Wool Industries Research Association*, Torridon Headingley, Leeds 6, England, proposes to publish a series of 11 volumes on wool research in which the results of 30 years of scientific investigation of wool and its processing have been incorporated

Feiga Fromiga, Helio Morganti (Brazil), J. F. Williams, J. C. Davies (West Indies), Mario A. Mascaro, Raul E. Daigo, Cesar Forn, Julio C. Gonzalez Maiz, Rafael Pedrosa Puertas, Jaun C. Salines (Cuba), Y. T. Chou (Formosa), W. L. McClerry (Hawaii), P. Honig (Java), K. C. Banerji (India), A. G. Keller (Louisiana), Louis Baissae (Mauritius), Jorge A. Aolano (Peru), M. A. Del Valle (Puerto Rico), J. L. du Tiot (South Africa), and Walter Scott (Venezuela).

### U.S. Technical Information Committee for Military Establishments

THE FORMATION OF A SPECIAL Committee on Technical Informa-

The first volume, published already, is volume 4 on "Carding" and will be followed by volumes 5 and 6 on "Combing and Tops" and "Worsted Drawing and Spinning" respectively. Volumes 5 and 6 are expected to be ready during this year.

### Grant for Calcutta University

THE GOVERNMENT OF INDIA have sanctioned a capital grant of Rs. 20 lakhs to the University in addition to the recurring grant of Rs. 2.19 lakhs every year.

### Announcements

*First International Congress of Biochemistry* — A new section entitled "Industrial Fermentations" has been included in the programme of the Congress to be held at Cambridge from 19th to 25 August 1949.

*Sir Ben Lockspeiser* has been appointed Secretary, Department of Scientific & Industrial Research, Britain, in succession to Sir Edward Appleton. He will be taking up his new duties in May.

*Dr. C. V. Raman* has been elected corresponding member of the French Academy of Sciences, Paris.

*Dr. Birbal Sahni*, Director, Indian Institute of Paleobotany, has been elected honorary President of the 7th International Botanical Congress to be held at Stockholm in July 1950.

*Grading of Wool* — The Indian Textile Division Council of the *Indian Standards Institution* has drawn up a Draft Indian Standard Specification for grading of wool for export. This has been done with a view to help the industry to grow on sound lines by standardizing the various grades of exportable wool and to ensure that the exported product conforms to those standards. The draft standard is being circulated

among wool producers, exporters and manufacturers and others. Comments on the subject will be received till the end of May 1949 by the Director, Indian Standards Institution, Block 11, Old Secretariat, Delhi 2.

*The Central Board of Irrigation Medal* — This medal has been instituted by the Board to encourage research relating to, and the technique and development of, water resources of India and all devices and matters connected therewith. The medal will be awarded every year for an outstanding original contribution of the preceding year, and will be awarded at the annual meeting of the Board. The contribution may be in the form of a paper presented to the annual meeting of the Research Committee of the Central Board of Irrigation or to any other engineering institution in India or may be an article

published for the first time in the *C.B.I. Journal* or any other technical journal published in India. Applications for the award, accompanied with 5 copies of the paper or article, should be addressed to the Secretary, Central Board of Irrigation, Kennedy House, Simla 4, to reach him before March every year.

*Russian Technical & Scientific Publications* — The Research Information Service, 509 Fifth Avenue, New York 17, N.Y., have introduced a new and much-needed service by cataloguing and preparing translations of articles from current Russian technical and scientific publications. The subjects dealt with are: petroleum, metallurgy, aeronautics, machinery, medicine and a few others. The publications can be had from the publishers at the above address (*Curr. Sci.*, 1949, 18, 53).

## ERRATA

### Vol. VIII, No. 3, Article: "Chemical Control for Power Alcohol Distilleries"

Page 91, R.H. col., item 8: for "12 to 13" read " $\frac{12}{13}$ "

" 92, R.H. col., item 20: for "Wash + Steam = Spent wash + Equilibrium vapour = Wash + Steam - Equilibrium vapour" read "Wash + Steam = Spent wash = Wash - Steam - Equilibrium vapour"

" 93, L.H. col., item I: for "gal. of spirit" read "gal. of proof spirit"

" 93, L.H. col., item V: for " $\frac{\text{Alcohol \% wash (9)}}{\text{Possible recovery (I)}}$ " read

"  $\frac{\text{Alcohol \% wash (9)}}{\text{Molasses \% wort (II) \times sp. gr. (5)}}$  "

" 93, L.H. col., item IX:

for " $\frac{\text{Alcohol made (24)}}{\text{Distilled wash (17) \times Alcohol wash (9)}} \times 100$ " read

"  $\frac{\text{Alcohol made (24)}}{\text{Distilled wash (17) \times Alcohol \% wash (9)}} \times 10,000$  "

" 93, R.H. col., item X: for "gal. of spirit" read "gal. of proof spirit"

" 94, R.H. col., item (f) 5: for "md./gal." read "lb./gal."

# Reports from States & Provinces

## BIHAR

### Mica Industry

THERE ARE ABOUT 600 MICA-mining centres in the mica belt of the province. Out of these a 100 are worked by mechanical means and the rest by manual labour. The average depth in the mechanized mines is 200'-250' and the deepest ones range from 600'-700'. The average depth of mines worked manually varies from 5'-50'.

About 15,000 labourers are employed in these mines and over a lakh are engaged in mica-processing factories. Mica-splitting is carried on as a cottage industry, over a lakh of women providing almost all the labour. About 250 firms hold licence under the *Bihar Mica Act* for mining and out of them 15 firms produce nearly 80 per cent of the total mica.

There are over 2,500 licensed dealers in mica in the province. The export of mica is in the hands of 60 exporting firms of which 8 are responsible for most of the export. The 2 biggest exporters of mica in Bihar are: *Messrs Chatturam Horliram Ltd.*, and *Messrs Chrestein Mica Industries Ltd.*, whose combined exports amount to over 50 per cent of the output. The mica industry is the chief dollar earner for the country. Small holdings, large number of small exporters and absence of any standardization of mica for export purposes are some of the chief factors which hinder the proper organization and expansion of the industry.

Over 50 per cent of the mica exports during 1948 was for the American market. The mica exports for 1948 are shown below:

Variety	Quantity Cwt.	Value Rs.
Block	8,497	64,30,626
Splittings	1,63,426	4,44,07,669
Ground and scrap	1,23,895	7,40,299

The serious threat to this industry in the shape of synthetic mica can only be countered by putting the industry on a sound footing by introducing improved methods of mining, standardization of material for export, better marketing practices, and by providing training facilities and amenities for the labour employed in the industry. (Contributed)

## WEST BENGAL

### Locomotive Factory

THE FOUNDATION-STONE OF THE first Indian locomotive factory was laid by the Minister for Railways and Transport during January at Mihijam in the Burdwan district. The factory, with an out-turn of 120 steam locomotives and 30 spare boilers annually, besides standardized spare parts, will be set up at a capital cost of Rs. 8,50,00,000. The workshop will cover an area of 3,000 acres and will be equipped with over 1,000 assembling and manufacturing machines. The total cost of machinery will be about Rs. 3,00,00,000. Besides, the foundry, furnaces and minor machine tools will be obtained in India at a cost of Rs. 1,00,000. The Maithon Dam across river Barakar, six miles from Mihijam, will supply the necessary power for the factory, which is expected to go into production in January 1950. Standardized locomotive parts will be produced during the first year.

## MADRAS

### Mineral Resources of Sandur State

THE STATE OF SANDUR NEAR Bellary is reported to have rich deposits of iron and manganese, according to a recent survey by the *Geological Survey of India*.

Smaller deposits of copper and gold are also reported. The extent of the deposits of iron and manganese ores are estimated at 2.5 and 170 million tons respectively. The report recommends the installation of pilot plants for the production of cast iron and ferro-manganese steel.

### Cotton Growing

THE *Indian Central Cotton Committee* has sanctioned a 5-year scheme for improving the yields of cotton varieties grown in the province, viz. "Northern", "Western", "Karunganni", and "Cambodia", and for trying out "Sea Island" cotton in Malabar and South Kanara districts. This scheme is expected to result in an increase in the cotton acreage in the province by at least 1 lakh acres. The area under cotton is expected to increase further by 1952, when the Lower Bhavani Project is expected to be complete.

## UNITED PROVINCES

### Sugarcane Development

TO IMPROVE THE YIELD PER ACRE of sugarcane in the factory zones of the province, the U.P. Government have inaugurated the Cane Development Competition Scheme which comes into force in the current cane season. The target is a minimum of 700 md. per acre which has been proved to be within the means of the average cultivator using approved methods of cultivation. The provincial Government has sanctioned a sum of Rs. 19,000 for this purpose.

The competition is held under 3 categories: (1) Among the cultivators — "Cane Development Zonal Competition"; (2) among the cane development zones in a range — "Inter-zonal Range Competition"; (3) among all the ranges in the province — "Inter-range Provincial Competition".

The province is divided into 4 ranges, viz.: (1) Western Range; (2) Rohilkhand Range; (3) Central and Banaras Range; and (4) Mideastern and Eastern Range. The varieties of cane recommended for cultivation in these areas are: Co. 313, Co. 385, Co. 421, Co. 353, Co. 453, Co. S. 76, Co. 356, Co. 386, Co. 527, Co. 456, Co. 395, Co. 393, Co. S. 109 and Co. 370.

# INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the *Gazette of India*, Part II, Section I, for March 1949.

## Inorganic Chemicals

40400. STEINSCHLAEGERL : Manufacture of carbon black: *Cracking carbonaceous material by mixing with hot gas.*
37641. THE NATIONAL SMELTING CO. LTD. : Removal of sulphur compounds from gases: *Contacting the gases with alkaline earth metal carbonate on a large porous support at 100°C. to 850°C. (Addition to No. 37640.)*
38945. SEAILLES : Method of extracting alumina from bauxite or similar aluminous ores and simultaneously obtaining a residue available as ore rich in iron: *Roasting bauxite with calcareous material, lixiviating roasted product to extract alumina, mixing residue left from lixiviation with further bauxite and repeating the cycle till the residue reaches desired iron content.*
38947. SEAILLES : Improved method of extracting magnesia from aqueous solution containing magnesium salts decomposable by lime and alkalis: *Reacting successively magnesium salts with lime and alkali.*
37623. DORR CO. : Calcining lime-bearing sludges: *Bringing solids and upflowing gaseous stream into calcining chamber under condition of suspending the solids, and calcining them to yield nodules of lime.*
39691. DE DIRECTIE VAN DE STAATSMIJNEN IN LIMBURG : Method of manufacturing a compound fertilizer from an acid solution which contains fluorine as well as calcium and phosphoric acid: *Maintaining the pH of the medium of the neutralizing and vaporization processes between 1.5 and 2.5.*
39937. I.C.I. LTD. : Improvements in ammonium nitrate compositions: *Maintaining a mixture of ammonium nitrate and potassium nitrate between 32°C. and 100°C.*

## Organic Chemicals

40084. STANOLIND OIL & GAS CO. : Process and catalyst for synthesis of hydrocarbons from carbon monoxide and hydrogen: *Contacting hydrogen and carbon monoxide with fluidized iron catalysts.*
39213. LES USINES DE MELLE : Saccharification of amylaceous materials in acid medium: *Through acid hydrolysis, neutralization, alcoholic fermentation and distillation.*
40072. N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ : Method of producing ethyl alcohol: *Passing a mixture of ethylene and water vapour over a catalyst comprising of diatomaceous earth pellets soaked with phosphoric acid at high temperature.*

## Food & Kindred Products

37242. LUDIN : Method of producing fat by means of micro-organisms: *Cultivation of yeast on carbohydrates and the extraction of the fat produced by a solvent.*

## Medical Research & Practice

37328. ELDERFIELD & HEAD : Anti-malarials: *Condensing 5, 6-dimethoxy-8-amino-quinoline with the hydrohalide of an alkylaminoalkyl halide.*
39032. ORTHO PHARMACEUTICAL CORP. : Production of compounds having vitamin A activity: *Subjecting cyclohexene derivative to hydrogenation, dehydration and acid or alkali isomerization.*

## Metals & Metal Products

39636. CARNEGIE-ILLINOIS STEEL CORP. : Electrodeposition of tin: *Electrolyte comprising acid solution of tin with an addition agent consisting of monobutyl-phenylphenol-sodium-monosulphonate or of diphenyl-p-phenylene-diamine or both and a sulphohone of phenol, cresol, resorcinol, or naphthol.*
38890. CARNEGIE-ILLINOIS STEEL CORP. : Electrolytic tin-plating baths and addition agents there for: *Addition agent for tin-plating bath consists of a polymerized reaction products of aniline or toluidine with acetone or methyl ethyl ketone.*
39302. THE MOND NICKEL CO. LTD. : Cast iron: *Graphitic cast iron containing retained magnesium.*
39498. CHAUDARY : Manufacture of metallic sheets, strips, rods and wires: *Roller rotated in molten metal which adheres to roller as scale or sheet which is passed between two smooth rollers.*

## Stone, Clay & Glass Products

37338. CHAMPMAN : Bricks: *Protruding portion of one brick, adjacent a mortar-retaining recess, projects into the adjacent recess of another brick.*

## Miscellaneous

40513. BRAKE & OSBORNE : Improvements in the treatment of water for the purpose of reducing the effects due to hardness: *Inducing electronic charges of high frequency from a valve oscillator having means for its frequency control.*

# Defence Science Conference

THE Defence Science Conference, organized by the Defence Ministry and inaugurated by Dr. S. S. Bhatnagar on April 20 at Delhi in the unavoidable absence of the Defence Minister, was the first of its kind to be held in India. It was intended to stimulate interest in defence science among Indian scientists, and to bring together research workers in defence establishments, universities and civilian research institutions so that problems of mutual interest may be discussed and ideas exchanged. Its success was largely due to the awareness on the part of scientists that co-operation with Government's plan for defence research would provide them opportunities not only to make their contributions to national security but also expand the field of their professional activities.

To appreciate the significance of the Conference, it would be useful to refer briefly to a few distinctive organizational features of scientific research in the Defence Department. Defence research has well-defined objectives and, therefore, lends itself to planning. It is essentially applied and developmental in character, and it caters to what Steelman\* describes as an "internal clientele". In other words, research in this field is intended primarily to meet the specific needs of the Department under whose auspices it is carried out. In this respect it differs from agricultural or medical research which caters to an "external clientele", that is, it meets the requirements, not of the sponsoring Department, but of the nation as a whole. If this feature of defence research is borne in mind, it will be clear that the agency which plans and guides research programmes should maintain intimate contact with the administration at the policy-making level, so that Service requirements may be precisely formulated and catered to.

The progress of applied or development work is dependent on knowledge revealed by basic or fundamental research, and significant results cannot be expected unless the frontiers of knowledge are deliberately and continuously widened. The Defence Department, keenly interested in the promotion of basic research, has to take steps to encourage such research activities in universities and civilian research centres where the "climate" for free inquiry and creative endeavour is favourable. In the United States of America, basic research in the Army is carried on largely outside the Department by contract, and applied and development work is the concern of the Technical Services and the Air Force. A similar distribution of research effort obtains in the United Kingdom. Thus, defence science is concerned with both basic and development research and the Defence Science Organization constitutes a bridge linking abstruse fundamental research at one end, and intensely specialized design, production, and proving work at the other. The Defence Department has a clear interest in the progress of research as a whole — in industry, the universities and in other Government Departments — and its manifest effort to enlist the understanding co-operation of the nations' scientists is necessitated both by enlightened self-interest and national security.

The need for sustained and adequate military research, and the need for increased emphasis on science in the training of officers for the Army, Navy and the Air Force have been widely recognized. Both these needs can be satisfied by securing a professional partnership between the officers of the Services and civilian scientists. Dr. Vannevar Bush, in his oft-quoted Report, *Science — the Endless Frontier*, points out how vital such partnership is for the promotion of national security, and stresses the need for a "permanent, independent, civilian-controlled

\* *Science & Public Policy*, 1947, III, 40.

organization, having close liaison with the Army and Navy, but with funds drawn from the Congress and with the clear power to initiate military research which will supplement and strengthen that carried on directly under the control of the Army and the Navy". The value of such an organization was proved during the last war, and Dr. Vannevar Bush was called upon to direct the activities of the war-time Office of Scientific Research and Development. Civilian-controlled organizations were in operation in several Allied countries during the war, and the integration of scientific and military thought secured thereby greatly influenced the course of the war. The view expressed by Vannevar Bush has the authority of experience and achievement and has been widely accepted.

Defence research, in any real sense, has yet to be initiated in India. Useful experience gained during the last war is, however, available. Development work started during the emergency when communications with Europe became difficult was concerned mostly with modifications of existing equipment and weapons to render them suitable for service conditions in India. Research was initiated on a small scale, and laboratories were set up. The expansion of the Air Force and the Navy called for new facilities for designing and testing, and posed new problems for solution. The emergency organization was inadequate to meet the requirements; it was necessary to shape it towards more enduring ends. Three years ago, the Government of India invited Dr. Wansborough-Jones, Scientific Adviser to the Army Council in the United Kingdom, to advise them on the setting up of a Defence Science Organization for India. More recently, the Government sought the advice of Prof. P. M. S. Blackett and of Sir Ben Lockspeiser. Plans have been prepared and

finalized, and a Defence Science Organization is beginning to take shape. In formulating its programme so that it may serve national interests effectively, a scientific approach is a desideratum. Science is required not merely to solve problems that are seen to exist; it is even more required to discover problems and to assess the order of their importance. Planning and programming demand scientific forethought and wise counsel, and in a matter so vital as national security, the understanding co-operation and collective wisdom of military and civilian personnel are essential. The organization of defence science in India is rendered particularly difficult on account of the overall paucity of scientific workers and meagre budgetary allotments. As pointed out by the Scientific Adviser to the Defence Ministry during the Conference, there is but one scientist for every 100,000 of the population, and the total research expenditure is hardly one-fiftieth of one per cent of the national income! Even so, the prospects for scientific development are bright in view of the country's prodigious, but yet untapped, resources both in talent and wealth, and the concerted measures which the Government, fully alive to the benefits of science, are taking to augment the man-power and material requirements for scientific research. There is enthusiasm among scientists to co-operate with Government in their plans for national security, an enthusiasm which was manifested in a clear manner in the discussions and declarations during the 3-day Conference. By holding the Conference, the Defence Science Organization has not only assured itself of the co-operation of Indian scientists; it has also succeeded in encouraging those fruitful contacts outside the "official channels" which provide the surest means of raising the morale and productivity of working scientists.

## Third International Conference on Forestry & Timber Utilization for Asia & the Pacific

**T**HE Third International Forestry and Timber Utilization Conference for Asia and the Pacific, convened by the Food and Agricultural Organization of the United Nations, was inaugurated by the Hon'ble Mr. Jairamdas Daulatram, Minister for Food and Agriculture, Government of India, on 28th March 1949 at the Cheluvamba Mansions, Mysore. Delegates from Burma, Ceylon, France, India, Malay, Netherlands, New Zealand, Pakistan, Philippines, Siam and the United Kingdom attended the Conference. Observers were present on behalf of Bhutan, Indonesia, Korea, Nepal, Portugal, *UNESCO* and *SCAP* (Japan). The Indian delegation was led by Sir T. Vijayaraghavacharya who was elected chairman of the Conference.

Mr. Norris E. Dodd, Director-General of the *FAO*, in his message, dwelt on the inter-dependence of the well-being of the land and the community, while M. Marcel Lelorp, Director of Forestry, *FAO*, explained the magnitude of the problems they had to face in Asia and the machinery needed to translate the recommendations of the Conference.

One immediate and concrete sequel to this Conference is the setting up of a regional forestry office at Bangkok with Dr. D. Y. Lin as its first Director. This office undertakes the collection and dissemination of information on all forestry matters between countries of Asia and the Pacific.

The general scheme of work for the Conference was on the same lines followed in the two earlier regional conferences to assess the forest assets and liabilities of each region, to estimate the gap between supply and demands for forest product, discuss remedies and to set up machinery to implement on a regional basis the recommendations of the Conference.

The Conference, after critical discussion, adopted resolutions on the following subjects : (1) Forest Resources ; (2) Forest Policy ; (3) Soil Conservation ; (4) International Control in Erosion Control ; (5) Forestry and Weather ; (6) Control of Grazing ; (7) Roving Agriculture ; (8) Large-scale Afforestation

and Reforestation ; (9) Domestic Needs ; (10) Increased Production ; (11) Balancing Supplies and Demand ; (12) Distribution of Timber ; (13) Standardization and Grading ; (14) Forest Inventories ; (15) Enumeration Methods ; (16) Minor Forest Products ; (17) Statistics ; (18) Research ; (19) Exchange of Information ; (20) Dissemination of Information ; (21) Training and Education ; and (22) Conservation of Wild Life.

The recommendations of the Conference fall into two main categories, viz. the immediate needs and the long-term requirements. While the Conference took note of the urgent issues raised by the former category and made valuable recommendations to meet them, even more valuable was its contribution to the long-term rehabilitation and development of the forest resources of the region. So much so, the first resolution of the Conference deals with "the Importance of Forest Resources", which reads as follows : "The Conference, recognizing that forests, as renewable, are essential to a country's agricultural life, industrial development, economic stability and national security ;

recognizing also, that forests and forest products, both major and minor, are essential to the life of the rural and urban population ;

calls the attention of the governments of all countries of the region to the need for dynamic policies for the conservation, development and utilization of forest resources according to the needs and possibilities of each country ;

declares that any failure by governments to accord due recognition to the great value of forest resources will have disastrous consequences for the economy of the country, both rural and industrial ;

urges governments to so manage their forests as to serve the rural and urban population and thus enlist the support of the masses for the implementation of a sound policy for the protection and development of forests ;

recommends that the attention of the governments of the region be drawn to the desirability of giving a high priority in their national, financial and economic plans



to the claims of forestry and the improvement of harvesting facilities."

*Forestry Commission for Asia* — The Conference considered that the conservation of natural resources and the provision of domestic needs in fuel-wood, charcoal and timber were the two outstanding problems of immediate urgency in many countries of the region. Solutions for these problems had also long-term implications. Bold schemes of protection and reforestation of the head-waters of great rivers were needed as also projects to combat soil erosion, programmes of afforestation and measures to effect flood control. Only thus could production and agriculture be made secure, water supplies be safeguarded, and living conditions be ameliorated. The effort required for these purposes has surpassed purely local or national interests. An international approach was necessary. The Conference, therefore, recommended the establishment of a Forestry and Forest Products Commission for Asia and the Pacific consisting of accredited representatives of all countries in the region, which should meet as and when necessary to co-ordinate matters of common interest to the region; to examine particular problems in the fields of forestry and forest products, both of immediate urgency and long-term significance; and make recommendations to governments through the Council of *FAO*.

It recommended, further, that in order to organize the work of this Commission, the Director-General of *FAO* set up a forestry and forest products working group in the region, composed of staff members of the Organizations Division of Forestry and Forest Products.

Of the other 23 resolutions passed by the Conference, 3 are of special importance to India.

The Resolution III deals with soil erosion which is a menace to most countries, and recommends the setting up, in each country, of a central authority for the co-ordination in both planning and execution, of land utilization policies. In this country the area

under forest is well below the safe minimum of about 25 to 30 per cent of the land area and it would be a short-sighted policy indeed if our effort at self-sufficiency in food should make inroads into our already inadequate forest areas — losing sight of the basic fact that forestry and agriculture are two different methods of utilizing the common capital of both, viz. the soil.

Resolution XVIII makes recommendations on the important subject of forest research. Attention was drawn to the fact that the existing research institutes, though well organized and well equipped, were extremely insufficient to serve the needs of the region and inadequately distributed geographically and there was lack of co-ordination of the research work carried out by different countries. India has, in the Forest Research Institute, Dehra Dun, one of the important forest research centres in this region, but it is not sufficient to cover even the needs of India. India's offer to co-operate fully by placing her available research facilities and experience at the disposal of the member nations at the Conference was very much appreciated.

The Indian delegation made positive contributions to the deliberations of the Conference on the subject of forest education which forms the subject-matter of Resolution XXI and which has a direct bearing on conditions prevailing in India at present. The facilities obtaining in India for forest education are good but grossly inadequate. The imperative need for trained foresters, forest training and research institutions and exchange of students and technicians was stressed. The type of specialist the country needs today is one with a grounding in the fundamentals of forestry and who is called upon not to manage forests but to run forest industries. The facilities for such specialized training are conspicuous by their almost total absence in India. One of the means suggested for achieving this is to provide a place for elementary forestry as a subject of liberal education in the curricula of universities.

# Characteristics of the Ionosphere over Calcutta ( March 1949 )

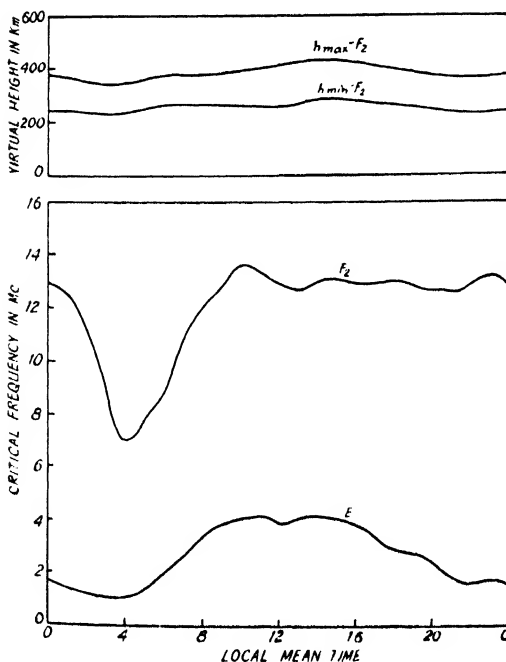
S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(Miss) T. K. PAL & A. P. MITRA

*Wireless Laboratory, University College of Science, Calcutta*

THE following are the ionospheric data observed at Calcutta for the month of March 1949. The observations were made at each hour of the day for 5 days a week.

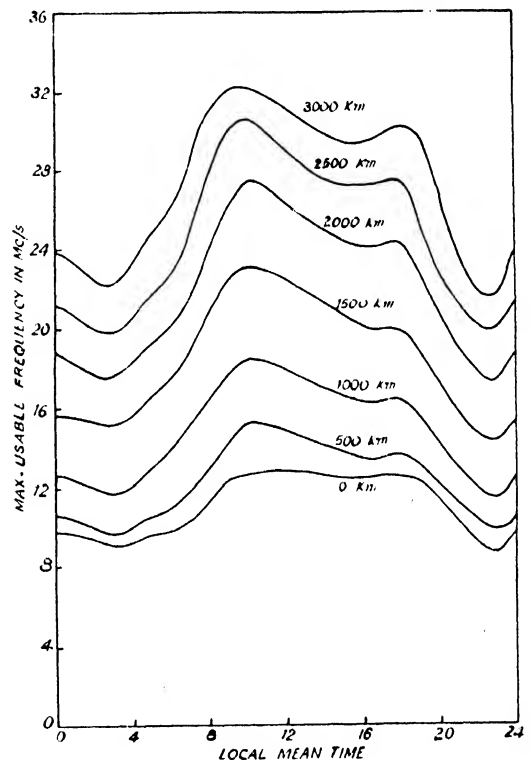
Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form during the month of March 1949. The figure represents the mean of both normal and abnormal values. Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmissions during June 1949 by reflection at the F region over Calcutta, based on the normal values of the data for March 1949. Table I gives the list of occasions when E region

ionization was found to be abnormal and the corresponding penetration frequencies and heights.



( 5 HOURS 54 MINUTES AHEAD OF G.M.T. )

FIG. 1 — MARCH 1949.



AT POINT OF REFLECTION.

FIG. 2 — PREDICTED M.U.F. FOR TRANSMISSION VIA  $F_2$  LAYER, JUNE 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f_o E_s$ Mc.	$h_{E_s}$ Km.
March 1949	1	04.00	2.50	95
		10.00	4.70	120
	2	09.00	4.10	120
		10.00	5.00	120
		11.00	5.70	135
		17.00	3.45	120
		18.00	3.30	120

TABLE I—*contd.*

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$h_{E_s}$ Km.
March 1949	3	18.00	5.00	135
		19.00	4.25	120
		20.00	3.20	120
		22.00	3.00	105
		23.00	2.70	105
	4	00.00	3.30	105
		01.00	3.20	105
		02.00	3.00	95
		03.00	3.00	95
		05.00	2.85	95
		16.00	4.60	120
		17.00	4.25	120
		18.00	4.25	120
	7	19.00	3.20	120
		20.00	3.50	120
		22.00	3.00	120
		23.00	2.70	120
	8	00.00	3.20	95
10		19.00	3.50	120
		23.00	3.15	120
11		00.00	3.50	120
		01.00	2.65	95
		02.00	2.50	95
		03.00	2.55	95
		04.00	2.45	95
15		22.00	4.25	120
		23.00	4.00	105

TABLE I—*contd.*

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$h_{E_s}$ Km.
March 1949	16	00.00	3.00	95
		01.00	2.90	95
		03.00	2.50	95
		07.00	3.55	120
	22	22.00	2.70	105
		23.00	2.40	105
	24	18.00	3.20	120
		19.00	3.20	120
		20.00	2.80	105
		22.00	2.60	105
		23.00	2.50	105
	25	22.00	2.65	105
		23.00	2.45	105
	26	00.00	2.20	95
		01.00	2.00	95
	28	18.00	3.15	120
		19.00	2.85	120
		20.00	2.50	105
		23.00	2.85	105
29		20.00	2.70	105
		21.00	2.50	105
		22.00	3.00	105
		23.00	3.00	105

## Patents & Designs Bill in the U.K.

THE BRITISH GOVERNMENT HAS JUST PUBLISHED the Patents and Designs Bill, recently introduced in the House of Lords. The Bill seeks to improve the patent law and thereby stimulate scientific and technical development in industry. It proposes to implement a large number of recommendations made by the expert committee, known as the Swan Committee, set up five years ago.

An important feature of the Bill is the provision of remedies for the abuse of patent rights or insufficient use of patented inventions. These provisions are directed not only against notorious types of abuse such as deliberate suppression of patented inventions but also cover cases where an invention is not being worked to the fullest possible extent, or where an export market is not being supplied or the working of another patent is hindered by the refusal of the

patentee to grant a licence on reasonable terms. Provision is also made for action by a Government Department in the public interest. There is no doubt that the Patents Enquiry Committee set up by the Government of India will carefully consider the feasibility of incorporating similar provisions in the Indian Patent Law.

The Bill embodies numerous other proposals of reform among which, to mention only a few, may be noted the extension of the grounds of opposition to check the grant of worthless patents, direction of all appeals from the Controller's decision to the Patent Appeals Tribunal, right of an exclusive licensee to take infringement proceedings in his own name and the manufacturer's right to obtain a declaratory judgement to resolve doubts whether a particular manufacture which he proposes to undertake would infringe a specified patent.

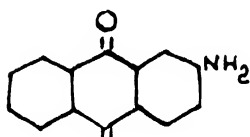
# German Dyestuff Industry\*

K. VENKATARAMAN

*(Continued from May issue)*

## APPENDIX 67

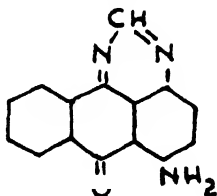
### 2-AMINOANTHRAQUINONE



In an autoclave (9 cu. m.) are introduced 2-chloroanthraquinone (1,400 kg.), ammonia 25 per cent (4,400 kg.), water (1,100 kg.) and arsenious acid 80 per cent (64 kg.). The autoclave is heated to 207°C. and a pressure of 45 atm. It is maintained at this temperature for 24 hr. At the end of the reaction the outlet valve is opened and ammonia absorbed in an absorbing vessel in the course of 8 hr. The gas is first passed through a Spitz cylinder, then 2 worm coolers and finally into an absorber in which water (500 l.) is placed. After the ammonia gas has been let off from the autoclave, it is filled with water (2 cu. m.), and the contents filtered. The filter cake is washed with warm water until there is no ammonium chloride in the filtrate. The filter cake is then dried when the crude amine (1,260 kg.) of purity 88 per cent is obtained. The yield is 87 per cent of theory.

## APPENDIX 68

### 4-AMINO-1 : 9-ANTHRAPYRIMIDINE



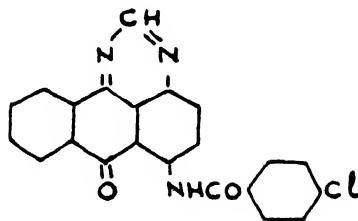
1 : 4-Diaminoanthraquinone, sublimed (0.5 kg.), ammonia 30 per cent (1.0 kg.),

formaldehyde 30 per cent (1.0 kg.) and Ludigol (0.375 kg.) are heated in an iron autoclave for 15 hr. at 88°-92°C. (pressure about 2 atm.). After cooling, the product is filtered, washed neutral with water and dried. Yield, 0.51 kg. of crude product.

The crude product is purified by dissolving in sulphuric acid, 96 per cent (5.0 kg.). The sulphate is then precipitated with the addition of water (2.5 kg.) in a dropwise manner at 100°C. After cooling, small needles of crystalline sulphate are filtered off and washed with sulphuric acid 58 per cent (2.0 kg.). The sulphate is pasted with hot water and decomposed with ammonia, filtered and washed neutral with hot water. Yield 0.42 kg. of pyrimidine = 84 per cent of 1 : 4-diaminoanthraquinone used or 81 per cent of theory.

## APPENDIX 69

### INDANTHRENE YELLOW 7GK POWDER (CRUDE)



The above dyestuff is prepared by benzoylation of Pyrimidine AFS with *p*-chlorobenzoyl chloride in nitrobenzene in presence of acid-binding agents like pyridine.

In an enamelled vessel provided with an agitator and an oil bath are introduced Pyrimidine AFS (60 kg.), nitrobenzene (300 kg.), pyridine (30 kg.) and *p*-chlorobenzoyl chloride (66 kg.). The contents are heated to 125°-130°C. and maintained at this temperature for 3 hr. The

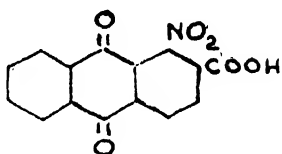
\* The documents mentioned in the report are available for consultation at Technical Information and Document Unit, German Division, Board of Trade, 40 Cadogan Square, London, S.W. 1.

benzoylation sets in at about 120°C. and finishes as a rule in 90 min. The contents of the vessel are maintained for a further 2 hr. at 125°-130°C. The vessel is allowed to cool without stirring and filtered. The filter cake is washed with nitrobenzene (200 kg.) till the filtrate is yellowish. The filter-pressed material is then freed from nitrobenzene and dried. From 60 kg. Pyrimidine, 90 kg. Indanthrene Yellow 7GK Powder (crude) are obtained, the yield being 91.2 per cent of theory.

For obtaining the dyestuff in paste form, it is dissolved in sulphuric acid, 96 per cent, and reprecipitated.

#### APPENDIX 72

##### 1-NITROANTHRAQUINONE-2-CARBOXYLIC ACID



In an acid-resistant 22 cu. m. vessel with lead stirrer is introduced a mixture of sulphuric acid 96 per cent (2,200 kg.) and sulphuric acid 58°Bé. (10,800 kg.). To this is added 1-nitro-2-methylantraquinone calculated at 100 per cent (640 kg.) in 2 lots. A warm solution of sodium dichromate (1,200 kg.) in water (640 kg.) at 50°C. is run in during 6 hr. The temperature is now raised in 6 hr. to 65°C. and the mixture heated at this temperature till no more of the carboxylic acid is formed (24 hr.). At the end of this period, 10-15 per cent of unconverted nitromethylantraquinone is present. Further oxidation serves no useful purpose as it takes a disproportionately long time. The reaction mixture is diluted with ice and water, cooled to 25°-30°C. and filter pressed. The filter cake is washed neutral to Congo paper with cold water.

The crude product is stirred in an alkali-resistant vessel of capacity 20 cu. m., provided with a stirrer, with 15 cu. m. water at 80°C. At this temperature caustic alkali solution 40°Bé (200-250 kg.) diluted with the same quantity of water is run in. The solution is tested with "Triacen" paper till it does not become red. A further quantity of caustic alkali (5-10 kg.) may be necessary. The mixture is stirred for 1 hr. at 80°C., filtered hot and washed with

warm water till the wash water does not contain the sodium salt of the desired acid. The filtrate and wash water are collected in an acid-resistant vessel of capacity 22 cu. m., and acidified with sulphuric acid, 96 per cent, (80 kg.) till the solution is acid to Congo paper. After brief stirring, the precipitated nitrocarboxylic acid is filtered, washed neutral with cold water and dried. Yield 560 kg. = 90 per cent of theory.

#### APPENDIX 74

##### INDANTHRENE BLUE RS

In a 5 cu. m. cast-iron vessel with stirrer are introduced 1,000 kg. caustic soda and caustic potash mixture of composition 67-68 per cent potassium hydroxide and 27-28 per cent sodium hydroxide and 200 kg. water-free sodium acetate. Then, at 180°C., in the course of 20 min., are introduced 500 kg.  $\beta$ -aminoanthraquinone (86-87 per cent) (= 436 kg. of  $\beta$ -aminoanthraquinone calculated at 100 per cent) from an automatic balance. After the addition is over a mixture of 60 kg. sodium nitrate, 40 kg. potassium hydroxide and 20 kg. sodium hydroxide, uniformly powdered, is introduced in the course of 2½ hr. The temperature of the melt should not rise above 200°-225°C. It is then stirred for half an hour and sucked off into a cylindrical vessel, 16 cu. m. capacity, containing 11 cu. m. of water at 40°C. The Indanthrene fusion vessel is then rinsed with 2 cu. m. of water. The temperature in the vessel rises to 60°-65°C., and it is cooled to 45°-48°C. in the course of 2 hr. by jacket cooling. It is then vatted by the addition of 750 kg. sodium hydrosulphite (14-15 per cent) under stirring. After allowing to stand for 2 hr., it is filtered on a 36-plate filter press. It is then washed with 1,600 l. water at 25°-30°C. and 150 kg. sodium hydrosulphite (14-15 per cent) and finally washed with water until the filtrate is not very blue but pale green. The filter cake is then blown out with air.

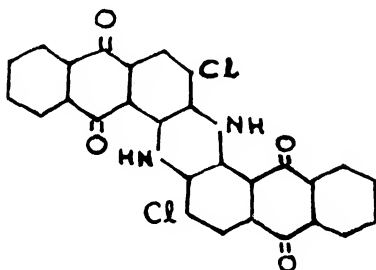
The filter cake is then introduced into a rubber-lined 8 cu. m. oxidation vessel provided with rapid stirring and containing about 1 cu. m. water and 20 kg. sodium hydroxide, and oxidized at 60°C. for 1½-2 hr. 2 lots (total about 490 kg. Indanthrene Blue RS calculated 100 per cent) are then combined and sucked off into a lead-lined cast-iron vessel of 24 cu. m. capacity or in a 14 cu. m. lead vessel, both provided with

stirring; for each double lot are placed in the vessel 4 cu. m. water at 60°C. and 160 kg. sulphuric acid 66°Bé. The slightly acidic dyestuff is then filtered off in a wooden press, washed neutral and dried (Indanthrene Blue RS Powder crude), or filtered in a rubber-lined Imperial filter and washed neutral.

The press cake thus obtained is reckoned as Indanthrene Blue RS, moist, calculated 100 per cent. Yield per lot 245 kg. Indanthrene Blue RS moist, calculated 100 per cent = 56 per cent of addition = 56.5 per cent of theory.

For the production of Indanthrene Blue RSN, the above product is pasted with 10 per cent Tamol solution and dried in 6-7 hr. The dried product is then finely powdered in a Raymond mill and mixed in a mixer with sodium carbonate and disodium phosphate and marketed as Indanthrene Blue RSN.

#### APPENDIX 74a INDANTHRENE BLUE BC



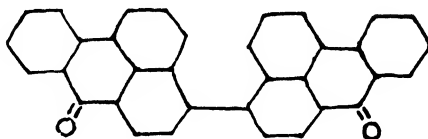
Indanthrene Blue RS is chlorinated in sulphuric acid (92 per cent), containing manganese dioxide, to the dichloro derivative of the azine. The filter-pressed material is then dissolved in oleum and reduced to dichloroindanthrone by the addition of anthracene disulphonic acid.

In an iron vessel of capacity 3.2 cu. m. fitted with a rapidly revolving stirrer are introduced sulphuric acid (96 per cent; 3,500 kg.) and Indanthrene Blue RS powder calculated at 100 per cent (350 kg.), and stirred till a complete solution is obtained. Water (175 l.) is now added in a thin stream, when the temperature rises to 50°-55°C. Finely powdered manganese dioxide (about 90 per cent purity; 17.5 kg.) is then added and the vessel closed. At 50°-55°C. chlorine gas (210-230 kg.) is led in at an hourly rate of 15 kg. When the chlorine content of the product reaches to 13.7 per cent, the product is filter pressed

and stirred with sulphuric acid 58°Bé (500 kg.) and diluted with water (400 l.). The sulphuric acid percentage of the mixture should not now go below 55°Bé. The product after dilution has a strength of 58°Bé. After standing for 40 hr., the product is filter pressed and filter cake washed with sulphuric acid 58°Bé (1,000 kg.).

The washed filter cake is now dissolved in a mixture of oleum (24 per cent; 2,000 kg.) and sulphuric acid (96 per cent; 500 kg.), the temperature not being allowed to rise above 350°C. Anthracene disulphonic acid (50 kg. calculated at 100 per cent) is now added and the temperature brought to 60°C. The mixture is kept till a test shows that the product is reduced; time taken is 10 hr. After cooling to 30°C., oleum (24 per cent; 300 kg.) is added and the solution precipitated under stirring in a 15 cu. m. vessel with warm water at 50°-60°C. The dyestuff is filtered and washed neutral with warm water. About 370 kg. Indanthrene Blue BC calculated at 100 per cent is obtained.

#### APPENDIX 75a 4:4'-DIBENZANTHRONYL

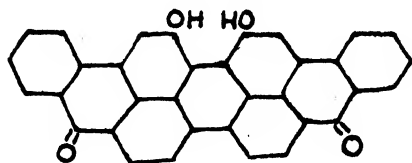


In a 3 cu. m. iron vessel provided with a stirrer are introduced pure isobutanol (385 kg. = 450 l.), powdered caustic potash (90 per cent; 500 kg.) and water-free sodium acetate (60 kg.). It is heated by closed steam to 140°-145°C. in about 2 hr. when isobutanol distils over and is returned from the condenser. It is stirred at this temperature for 1 hr. and cooled to about 100°C., when finely powdered sublimed benzanthrone (200 kg.) is introduced in the course of 1 hr. By the addition of benzanthrone the reaction mixture becomes rather viscous, so that the number of revolutions of the stirrer is reduced to 15-20 r.p.m. (Forset-Enor device). The reaction is exothermic, so that towards the end of the addition of benzanthrone the inner temperature is 112°C. It is stirred at this temperature for 1 hr. Then isobutanol (100 kg.) is added into the thick reaction mixture whereby the inner temperature falls to 105°C., and the fusion mixture becomes thinner, so that the

reaction mass can be stirred at the full speed of the stirrer. The reaction mixture is now cooled to 90°C. by water, cold water (1,000 l.) added, and by further cooling the temperature brought to 25°-30°C. The reaction mixture is then stirred for 30 min., and the lower 30 per cent aqueous potassium hydroxide layer is run off. In the vessel there is a deep-green paste which is a suspension of leucodibenzanthronyl in isobutanol. It is diluted with 400 l. water, and a solution of 80 kg. bleaching liquor in 600 l. water is run in, stirred for 30 min., and an azeotropic mixture of isobutanol with water is distilled over (inner temperature 90°-92°C.). This azeotropic mixture separates into 2 layers, the upper layer containing 83 per cent isobutanol and the lower layer 5 per cent isobutanol. The lower watery layer is run into the vessel till the inner temperature rises to 101°-102°C. The light-green mass is sucked off into a 22 cu. m. diluting vessel in which are introduced 2 cu. m. warm water. The vessel is rinsed with warm water and the diluting vessel filled with water to 8 cu. m., heated with open steam to 90°-95°C. and stirred at this temperature for 1 hr. It is then filtered through an iron filter press and washed with warm water. The pressed cake is dried under vacuum in an iron 3 cu. m. "Venuleth". Yield of dibenzanthronyl (82 per cent) is 200 kg. which is 82.3 per cent of theory.

#### APPENDIX 75b

##### 2:2-DIHYDROXYDIBENZANTHRONE



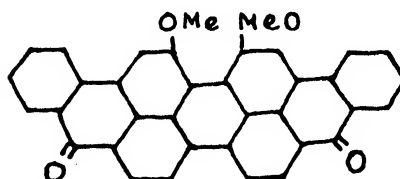
In a 3.2 cu. m. iron vessel provided with a stirrer are introduced 96 per cent sulphuric acid (2,000 kg.), 75 per cent sulphuric acid (2,500 kg.) and 4:4'-dibenzanthronyl 100 per cent (155 kg.). The mixture is stirred till the whole of the dibenzanthronyl is dissolved. After cooling to 15°C., pyrolusite (192 kg.) is added in 2-4 hr. in such a manner that two-thirds of it is added at 15°-25°C. and a third at 25°-35°C. through a 5 mm. mesh sieve. It is kept at 35°C. for 2 hr. and then filtered through an iron press.

The pressed cake (dihydroxydibenzanthrone and manganese sulphate plus

matrix) is stirred at ordinary temperature in an iron stirring vessel of 3.2 cu. m. capacity with monohydrate (3,000 kg.), till the dihydroxydibenzanthrone goes into solution. It is filtered through 2 resistant, revolving filters; each of 7 cu. m. capacity, and washed with 96 per cent sulphuric acid (3,000 kg.). The filtrate is diluted with water to 50°Bé in a lead diluting vessel of capacity 10 cu. m., cooled to 50°C., and filtered. The pressed cake in 3 portions is stirred in a resistant stirring vessel of 24 cu. m. capacity with water (15 cu. m.), and bisulphite solution 38°Bé (1,200 kg.) added. The mixture is boiled with open steam for 4 hr., filtered, washed neutral, and dried under vacuum at 130°C. Yield of dihydroxydibenzanthrone 100 per cent is 161 kg. (98 per cent of theory).

#### APPENDIX 75c

##### INDANTHRENE BRILLIANT GREEN FFB POWDER (CRUDE)



In an enamelled pressure vessel of capacity 3 cu. m. provided with a stirrer and an oil bath for heating is introduced trichlorobenzene (1,800 kg.). From another vessel provided with an agitator is introduced a suspension of caustic potash (200 kg.; dry content 99 per cent) in trichlorobenzene (250 kg.). The contents of the vessel are now heated to 180°-190°C. At this temperature is introduced from another vessel a suspension of dihydroxydibenzanthrone (150 kg.; calc. 100 per cent) in trichlorobenzene (300 kg.).

The vessel is now heated to boiling and maintained at this temperature for 1½-2 hr., when a mixture of trichlorobenzene and water (120-150 kg.) distils over. After cooling to 206°-208°C., methyl benzenesulphonate (300 kg.) is added in about 3 hr. in small lots. The contents of the vessel are now brought to the boil and maintained at this temperature for 3 hr.

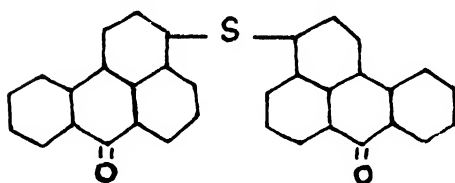
After cooling to about 180°C. the contents of the vessel are filtered off and the filter cake is washed with hot trichlorobenzene (1,000 kg.) at 180°C. The filter cake is

freed from trichlorobenzene and dried. Indanthrene Brilliant Green FFB Powder (crude) obtained is 141 kg., which is 94 per cent on the dihydroxydibenzanthrone used.

The dye (300 kg.) is made into paste form by dissolving in sulphuric acid (90 per cent; 4,500 kg.), immersing in cold water, filtering and washing neutral.

Trichlorobenzene used in the above reaction is recovered, about 100 kg. being lost in each batch.

#### APPENDIX 76 DIBENZANTHRONYL SULPHIDE

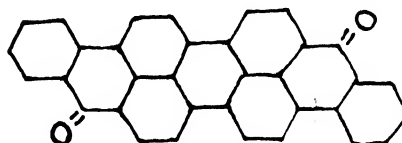


In a wrought iron autoclave of capacity 4 cu. m. for 15 atm. pressure with an elliptical iron stirrer and steam jacket, 1,420 kg. sodium sulphide solution 15 per cent (= 213 kg. sodium sulphide calculated 100 per cent) and finely powdered sulphur (45 kg.) are heated at 80°C. under stirring for 30 min. to bring the sulphur into solution, then cooled to about 30°C., and the operation carried on without stirring.

About 1,500-1,800 kg. bromobenzanthrone press cake (= 800 kg. bromobenzanthrone calculated 100 per cent) are added, while a week current of nitrogen is passed through. Now the autoclave is closed and to remove the air nitrogen at 1.5 atm. pressure is passed in and again released. Then the apparatus is filled with warm water up to 3.7 cu. m., closed again and the air once again displaced by introducing nitrogen. The mass is now heated to 135°C., whereby a pressure of 3.5-4.5 atm. is developed and stirred at this temperature for 40-50 hr. After cooling to 50°C., the mass is pressed into a wrought iron 14 cu. m. capacity stirring apparatus provided with "Balken" stirrer, containing 2 cu. m. warm water. The diluted content is filled into 12 cu. m. with washing water. After the addition of 70 kg. caustic soda 40°Bé the product is filtered in an iron filter press, and washed to neutrality with warm water (duration of washing is about 12 hr.). The filtrate and the washing flow into a vat containing waste sulphuric acid. The pressed mass

is dried in a vacuum drying cupboard with low pressure steam. Yield, 648 kg. of dibenzanthronyl sulphide dried = 102.2 per cent of theory.

#### APPENDIX 77 ISODIBENZANTHRONE



In a 3 cu. m. iron stirring vessel of capacity 3 cu. m. provided with a steam jacket are introduced isobutanol (600 kg. = 750 l.) and powdered caustic potash (90 per cent; 300 kg.). These are heated to boiling and maintained under reflux for 1 hr. (inner temperature about 140°C.). After cooling to about 70°C., finely powdered dibenzanthronyl sulphide (200 kg.) is introduced under stirring. The reaction mixture which is now at 60°C. is heated to 126°-128°C. whereby the alcohol begins to reflux. After about 2 hr. the reaction is complete.

A fusion sample diluted with water and vatted with hydrosulphite and alkali gives only a little dark filter residue. A yellow filter residue, which gives a blue solution with concentrated sulphuric acid and likewise a yellow outflow of filtrate from the filter, shows the presence of dibenzanthronyl sulphide.

When the correct test is obtained, pure isobutanol (100 l.) is pumped into the melt, cooled to 100°C., and water or water separated during isobutanol recovery (100 l.) added, whereby the temperature falls to 60°C. Then an azeotropic mixture of isobutanol and water is distilled over by heating the mixture to 90°-92°C., the lower watery layer of dilute isobutanol (5 per cent) being run back till the inner temperature is 101°-102°C. The dark-violet melt is introduced into a rubber-lined 14 cu. m. diluting vessel in which 8 cu. m. of water are placed, any substance remaining in the vessel being rinsed with water into the diluting vessel. This is heated with open steam to 95°-100°C., maintained at this temperature for 1 hr., and air blown through it without heating for 8 hr. The oxidation is complete when a sample on filtration does not give a red-violet filtrate. By the addition of cold water the temperature is

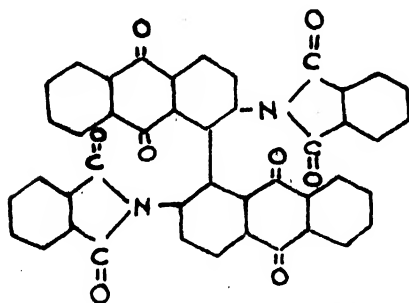


brought to 60°C., filtered through an iron press and washed well with warm water. The filter-pressed material is dried in an iron "Venuleth" under vacuum. Yield about 187 kg. of about 80 per cent isoviolanthrone (80.6 per cent of theory).

The watery isobutanol containing about 18 per cent by weight of water is recovered in a fractionating column. About 6 per cent of isobutanol is lost in the process.

#### APPENDIX 79

##### DIANTHRAQUINONYL PHTHALIDE POWDER (CRUDE)



It is prepared in 2 stages. In the first stage 1-chloro-2-aminoanthraquinone is condensed with phthalic anhydride in presence of ferric chloride, when water is split off, and 1-chloro-2-phthalimidoanthraquinone is formed. In the second stage 2 molecules of 1-chloro-2-phthalimidoanthraquinone are linked together by the action of metallic copper.

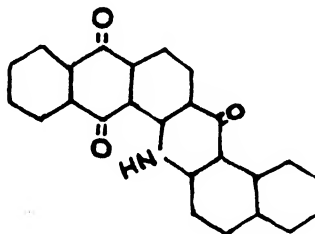
*Details of Process: First Stage* — In an iron 1.5 cu. m. pressure kettle, provided with stirrer and oil bath, is introduced first 170 kg. trichlorobenzene and, under stirring, 233 kg. 1-chloro-2-aminoanthraquinone 90 per cent (200 kg. at 100 per cent), followed by 132 kg. distilled phthalic anhydride and 4.4 kg. sublimed ferric chloride. The kettle contents are heated for 5-6 hr. at 215°-220°C. inner temperature, and for 1 hr. at 220°-230°C., whereby water and trichlorobenzene are distilled off. To facilitate the removal of water, a feeble stream of nitrogen is sucked through the kettle. After the attainment of 220°C. inner temperature, a minimum of 90-100 kg. of the mixture should be distilled (controlled through weighing). The melt is held at 230°C. for 5 hr. and cooled down to 100°C.

*Second Stage* — The melt is diluted with 300 kg. trichlorobenzene and heated again to boiling temperature (217°-220°C.) and

held for 3 hr. at the boil, during which a slow stream of nitrogen is maintained through the kettle. Water distils over mixed with trichlorobenzene such that in the stated time 100 kg. mixture are collected. The kettle is cooled down to 200°C. and by hand within 1 hr. are introduced 88 kg. copper powder. The mixture is heated to boiling temperature and maintained for 4 hr. Then the hot melt is diluted with 600 kg. trichlorobenzene, the kettle cooled to 130°C., and the product filtered in a closed filter press. The filter cake is washed with 700 kg. trichlorobenzene, and then dried in the Venuleth. Yield, about 459 kg. dianthraquinonylphthalide powder crude 48 per cent = 220 kg. calculated 100 per cent.

#### APPENDIX 82

##### INDANTHRENE RED RK POWDER (CRUDE)



1-Nitroanthraquinone-2-carboxylic acid is heated in *o*-dichlorobenzene with  $\beta$ -naphthylamine in presence of arsenic trichloride to give 1- $\beta$ -naphthylaminoanthraquinone-2-carboxylic acid, and this is cyclized by heating to boiling.

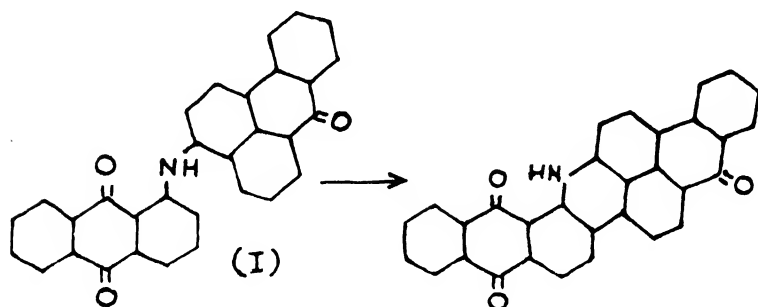
In an enamelled vessel of capacity 3 cu. m. fitted with a stirrer and an oil bath are introduced *o*-dichlorobenzene (3,000 kg.), nitrocarboxylic acid (calc. 100 per cent; 150 kg.),  $\beta$ -naphthylamine (162 kg.) and arsenic trichloride (33 kg.). Under stirring the temperature is raised to boiling in about 4 hr. (oil bath temperature about 235°C.). After about 3 hr., when the inner temperature is 155°C., the distillate is collected in a vessel of capacity 3 cu. m. in which a solution of caustic soda (20 kg.) in water (800 l.) is placed. When the inner temperature reaches 181°C., about 500 kg. of distillate is collected. The reaction mixture is now kept vigorously boiling (inner temperature 184°C.) for 3 hr., in which period for every 30 min. about 300 kg. of distillate are collected. The total distillate collected in 6 hr. is 1,800 kg. The reaction mixture

is then maintained for a further 4 hr. under slow boiling so that a very small quantity of distillate passes over (oil bath temperature about 205°-210°C., inner temperature 183°C.). The contents are now allowed to cool without stirring, blown into a filter press, and the filter cake is washed with *o*-dichlorobenzene (1,500 kg.). The filter cake is then freed from *o*-dichlorobenzene with steam, and dried.

The solvent is recovered from the distillate by separating from the aqueous sodium arsenate solution, and distilled in a vacuum pan with addition of soda (35 kg.). The dichlorobenzene lye from the filter press is treated with a third of its weight of concentrated sulphuric acid, stirred, and then with the addition of soda (12 kg.) distilled in a vacuum pan. Yield 151.5 kg. of Indanthrene RK Powder Crude calculated 100 per cent. The dyestuff is converted into a paste by dissolving in sulphuric acid (96 per cent), and precipitating with water.

#### APPENDIX 83

INDANTHRENE OLIVE GREEN B  
(PASTE)



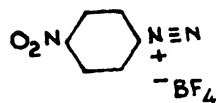
3-Benzanthronyl-1-anthraquinonylamine (I) obtained by the condensation of 3-bromobenzanthrone with  $\alpha$ -aminoanthraquinone is cyclized in isobutanolic potash to give the above dyestuff.

In a vessel of capacity 3 cu. m. provided with a stirrer and a steam jacket are introduced isobutyl alcohol pure (1,000 kg.) and caustic potash 90 per cent (150 kg.). By heating under stirring the temperature of the mixture is raised to boiling, and powdered (I) (200 kg.) is added together with isobutanol 200 kg. in about 30 min. In about an hour's time the reaction sets in which is shown by the thickening of the mixture. The contents of the vessel are

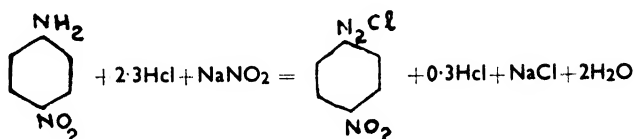
heated for 1 hr. more and then in 2 hr. isobutanol is distilled off. Cold water (700 l.) is now added and the inner temperature brought to 80°C. Isobutanol is now steam distilled for about 6 hr. The reaction mixture is then diluted with water, blown out with air, filtered at 60°C. and washed acid free. Yield of Olive Green B 100 per cent is 200 kg. About 85 per cent of isobutanol used is recovered.

#### APPENDIX 91

NITRAZOL CF EXTRA (PNA-QS = BOROF-  
FLUORIDE)



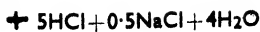
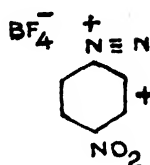
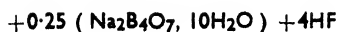
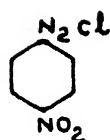
*p*-NITROPHENYLDIAZONIUM BOROFUORIDE  
Diazotization —



In a small diazotization vessel are introduced 32.5 l. water, 35 kg. ice and 203 kg. hydrochloric acid 30 per cent (= 60.8 kg. 100 per cent). To this, 22.5 per cent hydrochloric acid is added in about 3 hr., a mixture of 100 kg. *p*-nitraniline 100 per cent and 52.5

kg. sodium nitrite 100 per cent (5 per cent excess of the theoretically required amount). The temperature is maintained at 5°-10°C. The time for diazotization is 2½-3 hr. The mixture must always show the presence of nitrite and acid (direct test), and it is finally stirred for 2 hr. to complete the reaction, then cooled to 0°C. Then are introduced 1 kg. carboraffin dry and, after 10 min., 2 kg. kieselguhr. After stirring for 10 min. at 0°C., the mixture (360 l.) is filtered, and the residue washed with 35 l. water.

*Separation of Diazonium Borofluoride* — In the clear diazo solution are introduced 83 kg. borax Ia refined powder



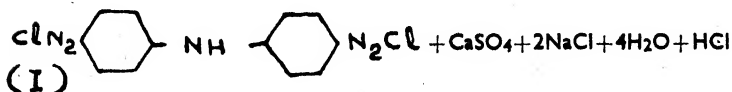
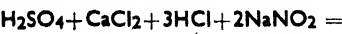
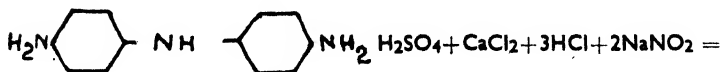
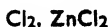
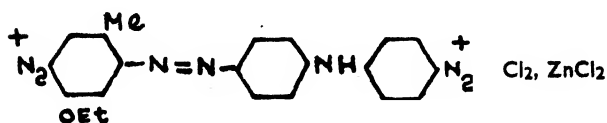
(= theoretical + 20 per cent) and 69.9 kg. hydrofluoric acid 100 per cent (= theoretical + 20 per cent) in 3 aliquots. The two are added alternately, beginning with borax, which, for the purpose of solution, is stirred for half an hour whereby the reaction remains acid. Before the hydrofluoric portion is run in, it is cooled to 0°C. After the addition of the last portion of hydrofluoric acid, the mixture is stirred for 2 hr., and cooled to 0°C. The volume is now 500 l. The precipitate is then filtered, washed with 37.5 kg. ice water and centrifuged. Yield, moist, 178 kg.

**Mixing & Drying** — The centrifuged diazonium salt is mixed with dehydrated Glauber's salt in the ratio of 2:1, and dried at 40°–45°C. for 36 hr.

**Standardization** — The standardization is carried out with alumina 23 per cent, and the balance with Glauber's salt and vacuum salt, so that the concentration corresponds to 22.5 per cent of the Fast Base. Yield is 95–96 per cent. In a small diazotization system, 300 kg. base 100 per cent can be handled.

#### APPENDIX 95

##### FAST BLACK SALT G (ESG—DS)

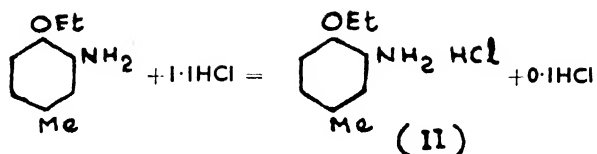


1. **Tetrazotization of p: p' — diamino-diphenylamine Sulphate** — In the large pre-warmed diazotization vessel are introduced 1,000 l. water at 80°C., and, then, under

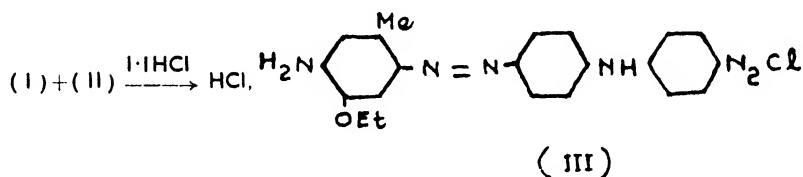
stirring 100 kg. diaminodiphenylamine sulphate moist 100 per cent (222 kg., purity 45 per cent), followed by 186 kg. calcium chloride lye (30 per cent by weight = 55.8 kg. 100 per cent). Hereby separates coarse crystalline calcium sulphate. At the end of the addition, the mixture is cooled to room temperature, under stirring, 184 kg. hydrochloric acid 30 per cent (= 55.2 kg. 100 per cent) are added, and cooled to 20°C. by brine. For the diazotization, the mixture is cooled to 0°C. by adding 447 kg. ice. At the beginning of the diazotization, about 25 l. of the base suspension are withdrawn. The total acidity reaches to: 1,000 kg. added water, 130.2 kg. water from calcium chloride solution, 73 kg. water from the base sulphate moist, 184 kg. hydrochloric acid, and 446.8 kg. ice water; the result being 1,834 kg. diluted hydrochloric acid of 3 per cent by weight. Here the sulphuric acid is not calculated as sulphate of the base. Then, under brine cooling, diazotization is carried out with 133.2 l. sodium nitrite solution 52.1 vol. per cent = 69.4 kg. 100 per cent = theory, diluted with 213 l. water (i.e. with 346.2 l. sodium nitrite solution 20 vol. per cent). The diazotization time is about 60 min. and the temperature 0°–10°C. The speed of diazotization is at first quick, and is then slow. Test for nitrite. The diazotization is complete when the test shows a yellow stain. No large nitrite excess is permitted, and any excess is now removed with the withdrawn sulphate of the base (25 l.). Finally, some nitrite solution is added so that there is a slight excess of nitrite. The volume of the diazo solution with foam = 2,600 l. This is filtered, and the filter washed with 2 × 250 l. water. The volume of filtered

diazo solution = 2,600 l.

2. **Preparation of Amino-p-cresol Ethyl Ether ("Kresaminathyl")** — In the dissolving vessel are introduced 500 l. water at



70°-80°C., and without stirring, 77.8 kg. Kresaminathyl 100 per cent (= 78.4 kg. of purity 99.2 per cent = theory + 2.5 per cent). After about 20 min. the Kresaminathyl liquefies. Now are run in under stirring 69 kg. hydrochloric acid 30 per cent (= 20.7 kg. 100 per cent = theory + 2.5 per cent). The base goes completely in solution. Carboraffin dry (1 kg.) is added, stirred for 15 min., diluted with 500 l. water, and the mixture filter pressed. The vessel and press are rinsed with 300 l. water. The volume of solution, now in the coupling vat, is made up to 2,950 l. at 5°-10°C. by about 1,000 l. water and about 500 kg. ice.



### 3. Coupling to form the Intermediate Product

The tetrazotized solution is, under stirring, introduced into the coupling vat. Time of addition is 15 min. and coupling temperature 5°-10°C. The diazo vessel is rinsed with 100 l. water. The coupling is feeble at the beginning, and in order to quicken the coupling, sodium acetate solution is run in under the surface of the liquid as described in (4). Time of running in 2 hr.; temperature 5°-10°C. The intermediate substance now separates.

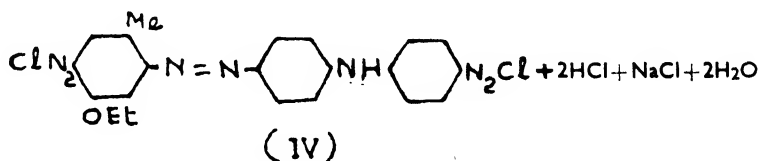
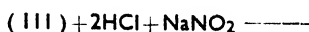
**Test**—A test with litmus paper should show no more red colouration (also no excess of tetrazo salt); it must give with diazotized *p*-nitroaniline solution a brown colouration (detection of excess of Kresaminathyl solution). After 4-5 hr. stirring the coupling is finished. In order that the intermediate product should separate as the hydrochloride, usually 96 kg. hydrochloric acid 30 per cent (= 28.8 kg. 100 per cent) and 400 kg. rock salt are added and stirred

for half an hour. Volume with foam = 6,500 l. Now the intermediate substance is collected in the filter press, and the coupling vat rinsed with about 1,000 l. mother liquor. The press is then blown dry.

4. *Preparation of Sodium Acetate Solution*—For 100 kg. diaminodiphenylamine sulphate 100 per cent are employed 213 kg. sodium acetate crystals dissolved in 600 l. water at 35°-40°C. under stirring, whereby all goes into solution and the temperature is 5°-10°C. The volume is now 7.5 l., and this sodium acetate solution is run in as described in (3). The dissolving vessel, after the removal of acetate solution, is rinsed with 50 l. water.

5. *Diazotization of Intermediate Substances (III)*—The intermediate substance (III) is removed from the filter press and suspended in the coupling vat with about 5,000 l.

water at 30°C. The volume is made up to 6,000 l. at 23°C. Then are added 123 kg. hydrochloric acid 30 per cent (= 36.7 kg. 100 per cent). After stirring for half an



hour, diazotization is effected with 69.9 l. sodium nitrite lye 52 per cent (36.4 kg. 100 per cent = theory + 5 per cent), diluted with 112 l. water, so that we have a 20 per cent nitrite solution. The diazotization temperature is 23°-25°C., and diazotization time 1 hr. The intermediate product goes into solution with a red-brown colour.

**Test for Nitrite & Acid**—The solution is stirred for half an hour after it shows the nitrite reaction. Hereafter are added 30 kg. kieselguhr. After stirring for 15 min. the mixture is filter pressed and washed with 600 l. water. The press is blown dry. Volume of filtrate 7,600 l.

6. *Separation of Zinc Chloride Double Salt*—The clear filtrate is treated with 76.4 kg. zinc chloride 100 per cent

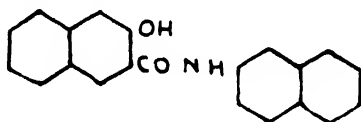
(= theory + 10 per cent) as lye of about 125 vol. per cent or dissolved in 100 l. water. The zinc chloride solution is run in very slowly. Volume 7,600 l. Then the whole is salted with 1,000-1,100 kg. vacuum salt (= 15 per cent of volume). The salting has to begin very slowly. After stirring for half an hour at 15°C., the product is sucked off. Yield, moist, is about 380 kg. (purity about 38 per cent; water about 28 per cent).

**Mixing & Drying**—The moist diazo salt is mixed in a Roland mixer with 220 kg. Glauber's salt R and 25 kg. zinc sulphate (anhydrous). Then it is dried on a Haveg sheet in a current of air for 48 hr. at 40°-50°C.

**7. Standardization of the Colour Salt to 20.5 per cent Purity**—The dried, purified diazo salt is mixed in a mixing machine for a short time, and then analysed. It is mixed with anhydrous zinc sulphate and Glauber's salt till a concentration of 20.5 per cent (about 22 per cent) of the base is obtained. This strongly diluted diazo salt is then powdered in a "Simplex-Perplex grinder". It is necessary to pulverize the salt very well and uniformly, 685 kg. Fast Black Salt G 20.5 per cent.

#### APPENDIX 96

##### NAPHTOL AS-SW



The raw materials used are pure toluene,  $\beta$ -naphthylamine, phosphorus trichloride and 2-hydroxy-3-naphthoic acid.  $\beta$ -hydroxy-naphthoic acid (250 kg.) is introduced in the reaction vessel, and over it toluene (1,800 kg.) is run in, and the two are stirred together. Under stirring is then added  $\beta$ -naphthylamine (190 kg.) and the contents of the vessel heated to 40°C. and stirred for 30 min. until all the  $\beta$ -naphthylamine is dissolved. The temperature is then raised to 75°C. in about 30 min., and at 75°-85°C. phosphorus trichloride (80 kg.) is added in the course of about 5 hr. At the end of this addition the temperature is raised to 107°C. in the course of 3 hr. and the contents maintained at 107°-111°C. for 20 hr. At the end of the reaction the

contents of the vessel are cooled to 40°C., and siphoned off into a distillation vessel, to which caustic soda (75 kg.) and water (1,000 l.) are added. The product is worked up as usual. From the above batch 396.7 kg. of product are obtained of purity 97 per cent, the yield thus being 95.3 per cent of theory. The m.p. of Naphtol AS-SW is 245°-247°C., and setting point 240°C.

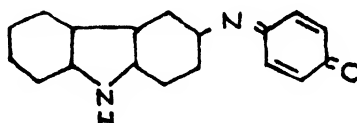
#### APPENDIX 106

##### *p*-NITROSOPHENOL

One adds in about 5 hr., at 0°C., an aqueous solution of 800 kg. phenol, 730 kg. caustic soda solution (33°Bé) and 698 kg. sodium nitrite (100 per cent) in 5,400 l. water to 6,400 l. of a solution of 1,586 kg. sulphuric acid (95 per cent pure) in water under stirring. The temperature is maintained at 0°C. by the addition of ice. The mixture is allowed to settle for 1 hr. and the supernatant liquid siphoned off. The residue is centrifuged and washed with ice water. Yield about 980 kg. paste = 840 kg. dry = 816 kg. 100 per cent pure.

#### APPENDIX 107

##### R BASE



In a 1,300 l. vessel, provided with a powerful agitator and cooling coils as well as jacket cooling, is placed 9,700 kg. of sulphuric acid (95 per cent pure), and cooled to 0°C. Herein is added 8 kg. "anthracene residue" and 800 kg. carbazole (about 98 per cent pure) in about 2 hr. After solution it is allowed to stand for 3-6 hr. and cooled to -28°C. In another vessel, provided with cooling coils, sulphuric acid (95 per cent; 4,300 kg.) is cooled to -10°C., and herein 592 kg. nitrosophenol, 100 per cent pure (= 670 kg. paste), is dissolved. After cooling to -27°C. the solution is transferred into the condensation vessel of 11,500 l. capacity. Under more powerful cooling, the carbazole solution cooled to -28°C., is added to the nitrosophenol solution in 2½-3 hr., so that the temperature is maintained at -23°C. (volume 9,200 l.). The indophenol solution is stirred afterwards

for an hour. An excess of nitrosophenol should always remain.

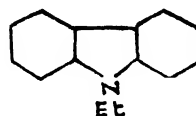
The reduction is carried out in 3 portions. A third of the indophenol solution is run into a lead tub of 30,000 l. capacity, provided with an agitator, in about 1 hr., to a mixture of 10,000 l. water, 3,000-4,000 kg. ice and 170 kg. of iron powder. During the time of addition 7,000-8,000 kg. of ice is added to maintain the temperature below 0°C. The temperature should be maintained close to -5°C. After additional 6 hr. of stirring, the temperature comes to 0°C. The reduction liquor is then green in colour. It is warmed to about 20°C. in about 2 hr. by means of steam, and then pumped at high pressure. The next day it is filtered through a filter press and washed with water until free of acid. The precipitate is once more treated with 12,500 l. of water and filtered. It is first washed with water, then with acidified water until free of iron. The paste from the whole batch (about 4,000-4,500 kg.) is dried on a cylinder with a steam pressure of about 4 atm. Yield 1,300 kg. dry (1,212 kg. 100 per cent pure; 92.4 per cent of theory).

*Hydron Blue RR* — The ingredients and the thionation process are the same as with Hydron Blue R. To distinguish Hydron Blue RR from Hydron Blue R, the finished dye paste is not acidified, but directly dried. The product is redder than the R mark, but is very variable in colour, tone and yield.

*Hydron Blue R (Powder)* — In the fusion pot, 1,710 kg. sodium sulphide ( $\text{Na}_2\text{S}$  conc.), 2,160 kg. sulphur and 1,800 kg. R base (leucoindophenol from carbazole and *p*-nitrosophenol) with 8,000 l. butanol are refluxed for 36 hr. The butanol is then distilled off in 6 parts. To each part in the distillation vessel are added 600 l. salt water and 1,200 l. distilled water, and one-sixth of the melt pumped in every 3 parts are transferred together to the desulphurizing vat, and desulphurized with 200 kg. sodium sulphide (crystals) for 1 hr. at 60°-65°C. The product is filtered through the press and washed free of sodium sulphide with 12°Bé salt water. The paste is then taken in about 12,000 l. distilled water, acidified with hydrochloric acid, stirred for  $\frac{1}{2}$  hr. at 50°C., filtered and washed with water until neutral. The paste is made up to about 20-22 per cent with water and dried to specification. Yield, about 4,250 kg. of the standard.

## APPENDIX 114

### N-ETHYLCARBAZOLE



In an agitated autoclave of 4,900 l. capacity, N-ethylcarbazole (1,800 kg.) and caustic potash (630 kg.) are heated to 150°-160°C., and carbazole (1,340 kg.) is then added. The autoclave is closed and heated in 6 hr. to 270°C. Water of reaction, some carbazole and secondary products pass over. The formation of potassium carbazole is now over. The temperature is allowed to fall to 215°C., and the valve leading to the receiver closed. Connection is established with an ethyl chloride cylinder containing 650 kg. ethyl chloride, which is heated to 10 atm. by means of a boiling water bath. The temperature falls to about 188°C., and in a run of about 16 hr. it is brought to 234°C. It is now allowed to cool to 160°C. in 8-12 hr., and excess ethyl chloride is blown over. After standing for 3 hr., during which potassium chloride is deposited, the supernatant base (1,600 kg.) is siphoned off into cold water, in which it solidifies. Then a further 1,600 kg. are siphoned off, and employed for the next operation. The residual potassium chloride is brought into solution in hot water, the base filtered through a filter press, and on cooling separated. This base is purified and then used as a solvent for the next batch. Yield, 1,533 kg. = 98 per cent of theory.

## APPENDIX 126

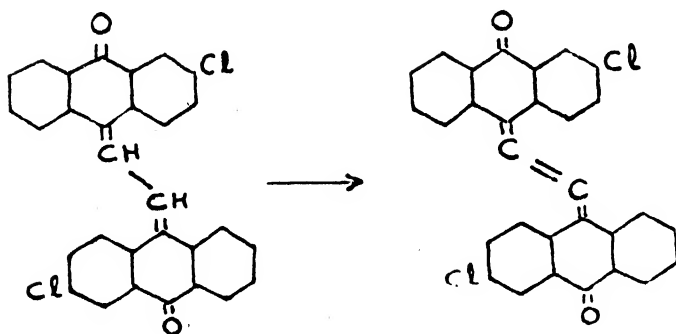
### 3-NITROCARBAZOLE

To dilute nitric acid from 1,600 l. water and 378 kg. nitric acid (40°Bé) in a stone-ware vessel with wooden stirrer, are added 300 kg. iron powder and finely powdered carbazole 100 per cent. The stirrer and vessel partition are rinsed with 200 l. water. The mixture is heated to 75°-80°C. with direct steam, and kept at this temperature for 4 hr. After addition of 1,000 l. cold water, it is filtered through a suction filter and washed neutral with cold water. The

product is dried at 50°-60°C. Yield, 370 kg. = 97.2 per cent of theory.

#### APPENDIX 129

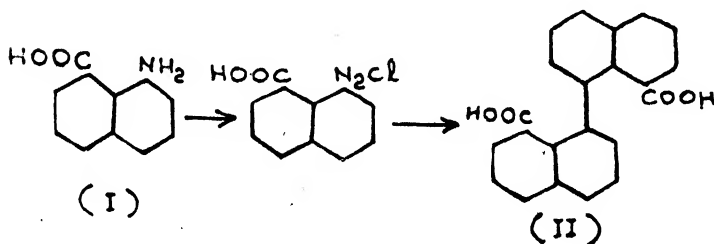
##### ANTHRONETHYLENE



In 1,200 l. of spirit and 104 kg. caustic potash, 120 kg. of anthronethane are quickly added at 75°C. and a current of air is passed in closed vessel through a disc of 2.2 mm. orifice for 6-7 hr. at 76°C. until a blue-end product is just perceptible under the microscope. Then it is filtered through a pressure filter and washed with spirit. 2 parts are stirred with 1,500 l. of water and 30 kg. of hydrochloric acid, filtered, washed and dried. Yield, 78 kg.

#### APPENDIX 133

##### 1:1'-DINAPHTHYL 8:8'-DICARBOXYLIC ACID



800 kg. of the acid (I) as paste are dissolved in 2 cu. m. water and 140 l. of soda lye; 61 kg. nitrite are added, and then a mixture of 450 kg. hydrochloric acid, 1,600 kg. ice and 1 kg. nitrite at 0°C. Neutralized with 800-900 l. soda solution, the diazo suspension is run into a mixture of 4 cu. m. water, 800 kg. ice, 150 kg. bicarbonate, a

cuprous chloride solution corresponding to 100 kg. copper, and 300 kg. ammonia at 15°-20°C. It becomes clear after the addition of 100 kg. ammonia and the acid (II) is precipitated from the filtrate with 450 kg. sulphuric acid, filtered, washed and dried in vacuum. Yield, 125 kg. as 100 per cent acid.

#### APPENDIX 137

##### PYRAZOLGELBKALI

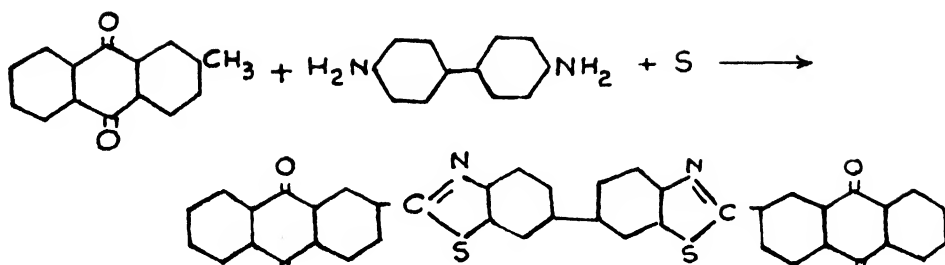
A mixture of 110 kg. pyrazoleanthrone and 215 kg. caustic alkali, prepared in a mixing drum, is mixed in a 1,000 l. fusion kettle with stirring with a solution of 5 kg. caustic alkali in 45 kg. spirit, and the kettle closed. The temperature rises through heat of reaction to 100°C.; it is then raised to 125°-130°C. and held at that temperature for 6 hr. The fusion is then complete. It is then blown under pressure at 110°C. and diluted slowly with 500 l. cold water. It is then run under pressure into the oxidation kettle (6,000 l.), adjusted to 330 l. at 60°C., oxidized with air, filtered and the paste hydraulic pressed (80 atm.). It is dried *in vacuo* at 80°C. Yield, 100-125 kg. dry; 89 per cent of theory.

#### APPENDIX 138

##### INDANTHRENE YELLOW GF

(a) *Crude Melt* — In a 1,000 l. melting vessel with ethyl carbazole bath are taken 500 kg. of lump sulphur at 225°C. Within 5 hr., through a funnel and screw conveyer, a uniform mixture prepared by grinding together of 120 kg. of  $\beta$ -methylantraquinone and 50 kg. benzidine are added. Temperature 225°-230°C. The inner temperature is kept for 2 hr. at 240°C., further for 2 hr. at 250°C. and finally 2 hr. at 260°C. At 260°-265°C. it is stirred for 3 hr. In the end the thick melt is drained out. The cooled melt is ground in a disintegrator.

(b) *Desulphurization* — In a 10,000 l. vessel, 5,100 kg. crystalline sodium sulphide are melted with indirect steam and brought to 100°C. In 1 hr. are added 1,830 kg.



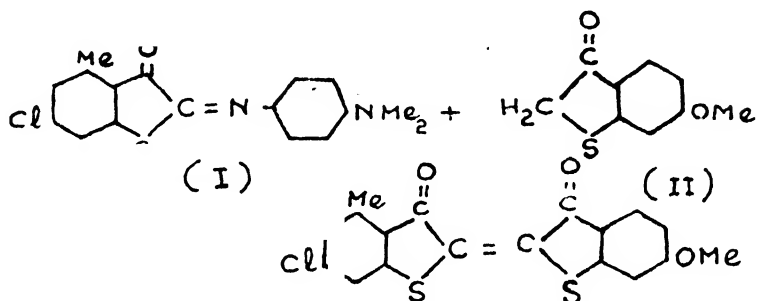
ground melt and stirred for 3 hr. more at 102°C., diluted to 9,000 l. and filtered at 80°-85°C. After washing with a solution of 150 kg. sodium sulphide in 1,500 l. water, and then with hot water till filtrate is colourless, the product is dried at 65°-70°C. Yield, 525 kg. crude dry dye.

(c) *Solution in Sulphuric Acid*—To 4,200 kg. 96 per cent sulphuric acid in a 5,600 l. vessel at 50°-100°C. are added in 1 hr. 525 kg. crude dye. Stirred for 4 hr. at 100°C., the solution is cooled to 20°C. and poured in 5,000 kg. ice and 9,000 l. water in 20 cu. m. stone vessel and stirred for 3 hr. Final temperature 10°C. Filtered through wooden press with nitrocellulose cloth and washed with cold water for 4 hr. The green paste weighs 2,100 kg. of which 21 per cent is obtained on drying.

(d) *Pure Paste*—The green paste is stirred in a vessel of 5,000 l. with 1,000 l. water for 5 hr. It must show a neutral reaction. Then it is poured in an alkaline stone chlorinating vessel of 22 cu. m. under stirring. According to experience the necessary hypochlorite liquor = 3,500 l. (14 per cent chlorine) and caustic soda to bring caustic soda content to 2.5 per cent are added. Stirred for 4-5 hr. and allowed to remain overnight, then heated to 100°C. with direct steam. When chlorine solution is not sufficient, more is added till potassium iodide paper after 1 hr. still gives the best. Mass is diluted to 15,000 l. and filtered through wooden press, and washed with hot water till the washing are neutral to turmeric. Pure orange wet paste, 1,700 kg. = 440 kg. dry.

#### APPENDIX 140

##### INDANTHRENE SCARLET B



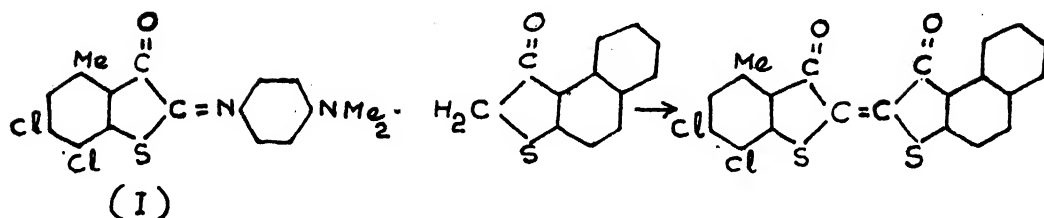
In a double-walled, enamelled vessel are introduced dry chlorobenzene (1,600 l.), "rosa-anil" (I) (132 kg.), methoxythionaphthene (II) (79 kg.) and piperidine (8 kg.). These are heated to 115°C. and maintained for 2 hr. at 115°-118°C. At this stage, condensation to an insoluble dye takes place. The dye is filtered off at 50°C. and washed thrice with 350 l. of warm chlorobenzene (50°C.) till the filtrate is light in colour. The filter cake is freed from chlorobenzene by steam and dried at 70°-80°C. The yield is about 190 kg. of standard dyestuff.

#### APPENDIX 142

##### VAT BROWN IVD

The "anil" (I) (114.5 kg.), 2:1-naphthoxythiophene (60 kg.) and piperidine (6 kg.) are stirred in dry chlorobenzene (1,350 l.), and heated to 115°C. The temperature is maintained at 115°C. for 2 hr., and then cooled to 70°C., and the product filtered. The filter cake is washed thrice with 300 l. warm chlorobenzene each time, freed from chlorobenzene with steam, and dried. Yield about 160 kg. paste or 113 kg. dry powder.



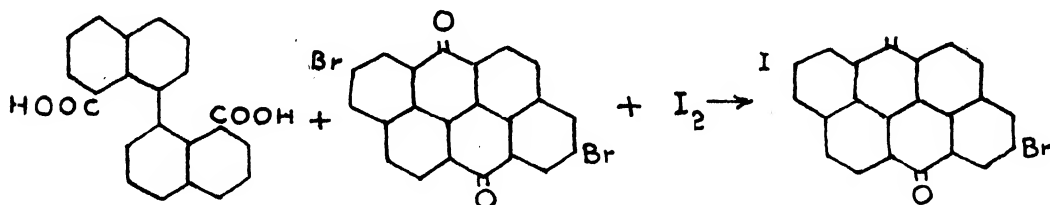


## APPENDIX 147

## VAT SCARLET RM

Dinaphthylidicarboxylic acid (89.4 kg.), dibromoanthrone (80 kg.), and arsenic acid (101 kg.) are dissolved in monohydrate (1,600 kg.) at 45°C. in the course of 1 hr., and diluted with water (16 l.) when the

red colouration, which then turns green. The contents of the vessel are allowed to remain overnight. Then, under cooling, oleum 65 per cent (490 kg.) is run in at 35°C. After cooling to 25°C., iodine (1.3 kg.) and bromine (90.5 kg.) are added. The contents of the vessel are now heated and agitated. In the course of an hour the temperature is

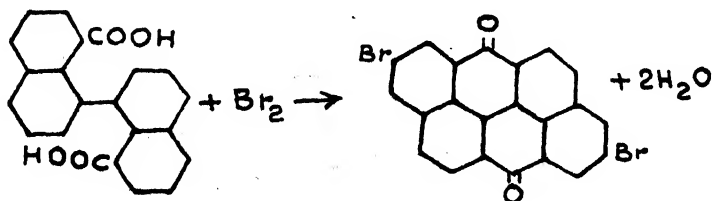


temperature rises to 60°-70°C. Next day iodine (85 kg.) is introduced and the contents heated to 132°C. in 2-3 hr. and maintained at 132°-135°C. for 5 hr. It is then cooled, and with the addition of water (400 l.) the sulphuric acid concentration is brought to 85 per cent. It is now filtered and the filter cake washed 5 times with 85 per cent sulphuric acid (200 l. each time). The filter cake is agitated with 2,000 l. water at 45°C. for 4 hr., refiltered and washed free from acid. The filter cake is now stirred for 1 hr. with warm water (2,000 l.) at 80°-90°C. and soda lye (240 kg. 33°Bé). After this it is filtered and washed. The yield is 550-650 kg. paste = about 195 kg. dry = 86.2 per cent of theory.

## APPENDIX 148

## INDANTHRENE BRILLIANT ORANGE RK

In an enamelled vessel of capacity 2,000 l. are introduced monohydrate (1,550 kg.) and 1:1'-dinaphthyl-8:8'-dicarboxylic acid (188 kg.) under cooling. Solution takes place first with a

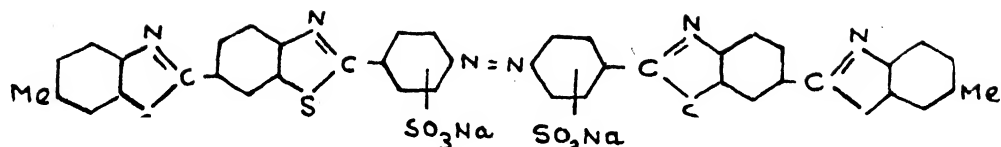


brought to 30°C., and then more slowly to 65°C., where it is maintained for 3 hr. After cooling to 50°-55°C., warm water at 60°-70°C. (450 l.) is run in. The contents are now filtered off and washed neutral. By the introduction of water the sulphate are hydrolysed and light orange-coloured dibromoanthrone results. The dyestuff obtained in paste form is 750-800 kg., the yield being 90 per cent of theory.

## APPENDIX 153

## SIRIUS LIGHT YELLOW RT

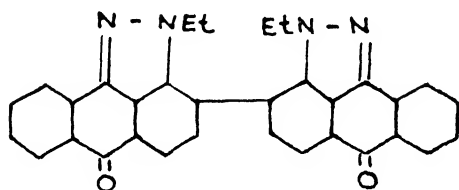
487 kg. of primuline acid (m.w. 373) are suspended as paste in 2,000 l. of water, and dissolved with about 270 kg. of soda lye solution of 33°Bé. It is diluted with water and ice to 8,000 l., and at 3°C. 115 kg. sodium hydroxide lye of 33°Bé and, then, with good stirring, 2,400 l. of sodium hypochlorite



(224 kg. active chlorine) are added in 6-8 min. The temperature rises thereby to 7°-9°C., then slowly to 15°-17°C. After standing overnight, excess chlorine smell should faintly persist; otherwise, a little hypochlorite solution is added. The dye is salted out with rock salt 650 kg. as a 11°Bé solution. Yield, about 780 kg., standardized at about 1,780 kg.

#### APPENDIX 154

##### DIETHYL RED (INDANTHRENE RUBINE R)



120 kg. "Pyrazolgelbkali" are ground with 200 l. chlorotoluene for 18 hr. in a flint mill, and discharged into the alkylation kettle. The mill is rinsed with 900 l. chlorotoluene. Total charge = 1,000 l. = 1,150 kg. chlorotoluene. Then are added 112 kg. ethyl *p*-toluenesulphonate and 12 kg. powdered caustic alkali. The mixture is heated at 125°-130°C. for 9 hr. when alkylation is complete. Then it is filtered hot. The cake is washed, stirred thrice with 200 l. chlorotoluene at 100°C., filtered off, and finally washed into the suction apparatus with 100 l. chlorotoluene and sucked dry. The adhering chlorotoluene is steam distilled. The dye is filtered and washed neutral with water. Yield about 140 kg. wet or about 80 kg. dry.

The filtrate of chlorotoluene is distilled with steam; yield about 95 per cent of the used chlorotoluene.

## A Method for the Preparation of Dioctyl Sebacate

DIOCTYL SEBACATE HAS COME INTO USE AS a plasticizer<sup>1</sup> as it imparts not only low temperature flexibility to polyvinyl chloride and its copolymers, but also possesses non-volatility characteristics obtainable with dicapryl phthalate or dibenzyl sebacate. Methods for the preparation of this plasticizer are not reported in literature. A method employing *p*-toluene sulphonic acid as catalyst has been worked out in these laboratories. The usual catalysts—conc. sulphuric acid and dry HCl gas—gave poor yields. The method of preparation is briefly as follows:

Sebacic acid (10 parts), secondary octyl alcohol (13 parts), and *p*-toluene sulphonic acid (1 part) were dissolved in benzene in a round-bottom flask and the mixture refluxed for 20 hr. The water formed was removed through a trap. The reaction mixture was extracted with ether, the

etheral solution washed with dilute sodium carbonate solution and dried over anhydrous sodium sulphate. After removal of ether, the residue was distilled under 5 mm. pressure and the fraction, boiling between 255°-257°C., collected. A yield of 34.5 per cent pure dioctyl sebacate on the weight of sebacic acid was obtained. The product is a colourless, odourless liquid; sp. gr. at 15°C., 0.920; sap. val., 258; acid val., 0.4; ref. index at 20°C., 1.4498.

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KARIMULLAH

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Council of Scientific & Industrial Research  
Delhi*

#### REFERENCE

1. *Rubber Age*, 1945, 56, 633.

# A Study of the Colouring Matter of *Tambul* Seeds\*

T. R. SESHADRI

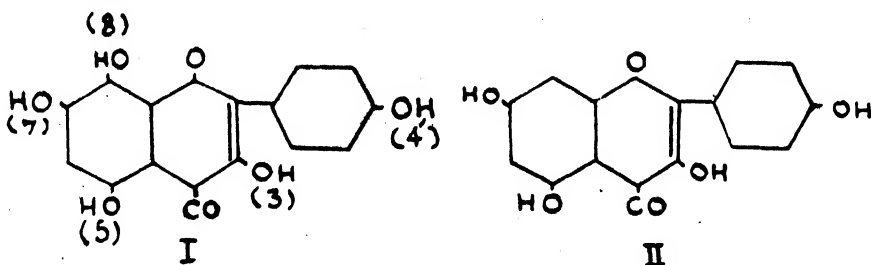
Andhra University, Waltair

**F**LAVONES and flavonols forming the most important group of anthoxanthins were originally valued highly as mordant dyes. In this respect they have been almost completely displaced by synthetics. More recently, however, their physiological properties have been attracting attention<sup>1</sup>. A precise knowledge of these yellow colouring matters is useful in other directions also. For example, it gives a deeper insight into the botanical classification of plants. As the result of the endeavours of chemical investigators in various countries, considerable progress has been recorded not only in the discovery of new types of compounds, but also in the development of newer and more efficient methods of studying their molecular structure and effecting their synthesis which will lead ultimately to a comprehensive picture of their evolution and function.

Hydroxy flavones and flavonols occur free in a number of cases. But it is more common to meet with them in a combined form. One or more of the hydroxyl groups are protected either by glycoside formation or by methylation or methylenation. Exact knowledge as to why these modifications are needed in the plant is not yet available. But it could be safely stated that factors relating to solubility, stability to oxidation and light absorption are involved. When there are a number

of hydroxyl groups in a molecule capable of protection in the above manner, the exact position or positions involved is of some importance because considerable variation in properties can arise thereby. Methods of locating these positions have been explored continuously and marked success has been achieved during the past few years. The study of the colouring matter of *tambul* seeds provides an example of such work on partial methyl ethers.

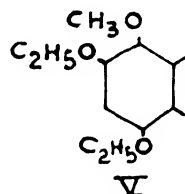
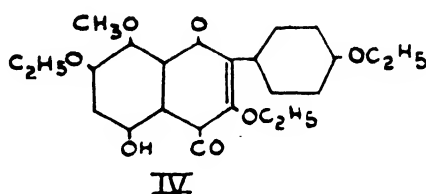
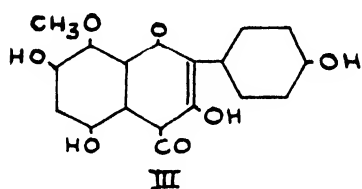
It is better to start with the discovery in 1935 of herbacetin (I) by Neelakantam and Seshadri<sup>2</sup> in the Indian cotton flowers, *Gossypium herbaceum*. The constitution of the new flavanol was soon determined at Waltair<sup>3</sup> and confirmed by synthesis at Oxford<sup>4</sup>. But there was considerable difficulty in isolating it from the cotton flowers, because it occurs along with 3 other flavonols, gossypetin, quercetin and kaempferol, partly free and partly as glucoside. The method of synthesis also was not easy at that time though a considerably simpler one has recently been discovered<sup>5</sup>. In order to supply other workers who were in need of small authentic samples, a search for more convenient sources was made. The flowers of *Thespesia populnea*<sup>6</sup> yielded herbacetin, but it was again associated here with kaempferol and the question of separation existed. Bose and Bose<sup>7</sup> subsequently reported the



\* Paper presented at the Symposium on Plant Products held during Silver Jubilee Celebration of the Indian Chemical Society (January 1949).

isolation in a very small yield (0.006 per cent) of tambulin from *tambul* seeds which form a well-known Indian drug and contain a sweet-smelling essential oil. This was shown to be a trimethyl ether which could be demethylated to herbacetin. Since the seeds were readily available and no difficult separations were involved, they appeared to form a satisfactory source for the quick preparation of small samples of herbacetin. We<sup>8</sup> were actually successful in obtaining much better yields (0.03 per cent) of the colouring matter than previous workers. Surprisingly, however, it was not tambulin, but a new chemical

But in cases where there are a number of free hydroxyl groups, this procedure has not worked satisfactorily. Neither does the decomposition take place well nor is the identification of the fission products easy. Consequently, in the study of partial methyl ethers, we have been adopting the practice of protecting these free hydroxyls by ethylation and then subjecting the mixed methyl ethyl ether to fission. By this means not only the decomposition of the flavonol molecule and identification of the products are rendered smooth and precise, but even confirmation by synthesis becomes comparatively easy.

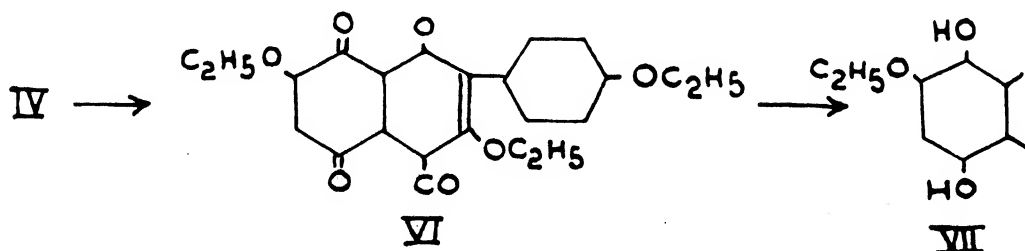


entity which we named tambuletin. It was a monomethyl ether yielding herbacetin on demethylation and thus our original object of preparing this flavonol was attained.

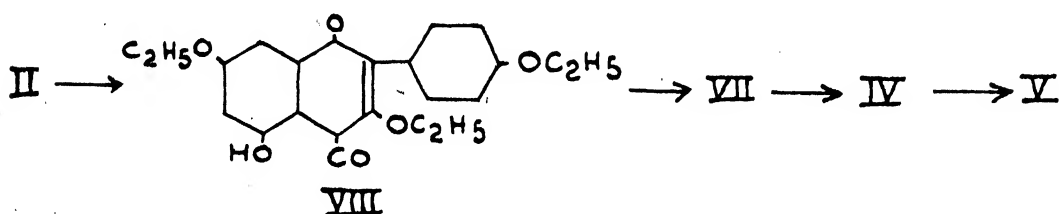
Next the chemical constitution of tambuletin was investigated in detail. Its properties and colour reactions indicated that it had the methoxyl group in the 8- position (III). It differed markedly from herbacetin (I) and resembled closely kaempferol (II) in the reactions with dilute alkali, alcoholic ferric chloride and *p*-benzoquinone. It could, therefore, be concluded that the hydroxyl in the 8- position was immobilized by ether formation. Though these colour reactions are very useful for purposes of diagnosis, errors have arisen in the past by depending too much on them. Confirmation by more definite methods had to be sought. It was usual to subject the new substance (partial methyl ether) to fission with alkali and identify the products of decomposition.

Actually in the case of tambuletin<sup>9</sup> though ethylation was carried out, there was no need to resort to alkali fission. Simpler methods whose usefulness had been fully established by our earlier work were adopted. Tambuletin could be ethylated in two definite stages yielding a tri- (IV) and a tetra-ethyl (V) ethers. The former exhibited the reactions for a free hydroxyl in the 5- position and was oxidized readily with nitric acid yielding a quinone, reduction of which produced a quinol. The final product was found to be identical with a synthetic sample of 3 : 7 : 4'-triethoxy-5 : 8-dihydroxy flavone (VII). It could, therefore, be concluded that the oxidation of *o*-triethyl-tambuletin involved demethylation and this could happen only if the methoxyl group was present in the 8- position. The transformations involved were represented as follows.

More conclusive evidence was provided by the synthesis of the tri- (IV) and tetra-ethyl



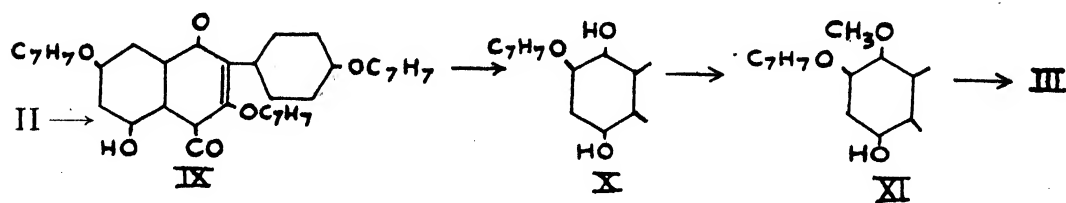
(V) ethers of tambuletin. This work as well as the synthesis of the quinol (VII) could be readily accomplished by the application of the new discovery of fascile nuclear oxidation in the flavone series<sup>15</sup>. Kaempferol (II) was the starting point; it was subjected to partial ethylation to form *o*-triethyl-kaempferol (VIII). Oxidation with alkaline persulphate yielded quinol (VII). Partial methylation with dimethyl sulphate and anhydrous potassium carbonate in acetone solution converted it into the 8-methyl ether, the resistant 5-hydroxyl being left out. This was identical with *o*-triethyl-tambuletin (IV). Subsequent ethylation yielded 8-monomethyl-3:5:7:4'-tetraethyl ether of herbacetin which was identical with *o*-tetraethyl tambuletin (V). The course of the various stages in the synthesis had been well established earlier by detailed study of simpler examples.



The synthesis of tambuletin<sup>10</sup> itself was carried out as the next stage. Here again kaempferol was the starting material and nuclear oxidation and partial methylation were employed. But some of the hydroxyl groups had to be protected by partial benzylation and the benzyl groups removed finally. There was some difficulty in the benzylation and in the oxidation of the benzyl ether. But with adequate precaution the reactions could be accomplished satisfactorily.

to be solved. From their study of tambulin Bose and Bose<sup>7</sup> proposed that it was 3:8:4'-*o*-trimethyl herbacetin (XII). We could prepare a substance of this structure starting from 3:4'-dimethyl ether of kaempferol (XIII) and passing through the stages indicated below<sup>11</sup>.

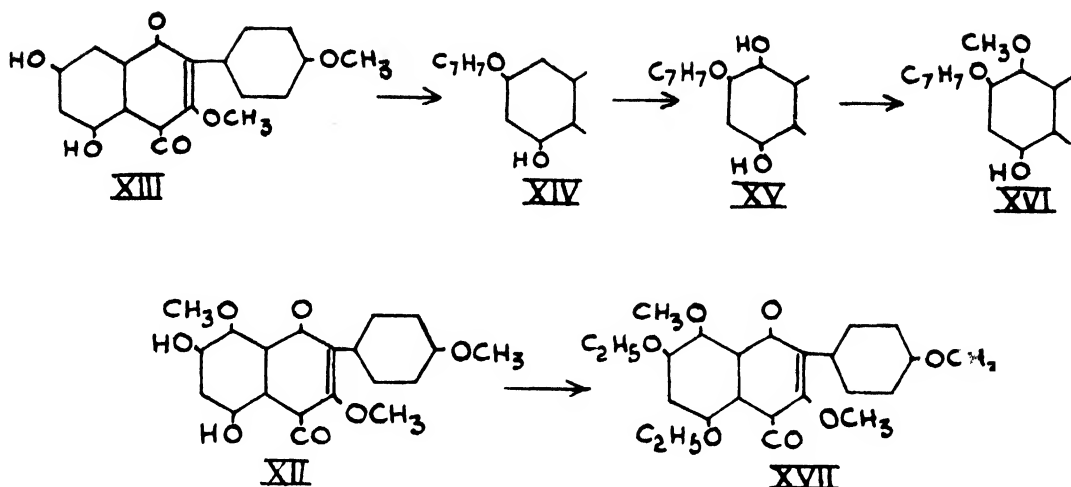
The synthetic product differed markedly from tambulin. Its melting point was much higher and so also was the melting point of its acetyl derivative higher than that of tambulin acetate. That the structure of the synthetic substance was correct was confirmed by conversion into its diethyl ether which was found to be identical with a synthetic sample of 3:8:4'-trimethoxy-5:7-diethoxyflavone (XVII). Consequently the constitution of tambulin had to be revised. Bose and Bose showed that a methoxyl was definitely present in the 4'- position because



It is rather surprising that in the number of extractions of *tambul* seeds that we carried out, only tambuletin was isolated and no tambulin as originally reported by Bose and Bose<sup>7</sup>. This is a puzzle which remains still

anistic acid could be isolated as a product of fission with alkali. Since the compound failed to reduce alkaline *o*-dinitrobenzene or chloropentamine cobaltichloride, there was proof that it did not have *ortho* or *para* dihydroxy groups. It was, therefore, concluded that a methoxyl was in the 8- position. This was also justified by analogy with tambuletin which has a methoxyl in the same position. They considered that the third methoxyl was in the 3- position, since the

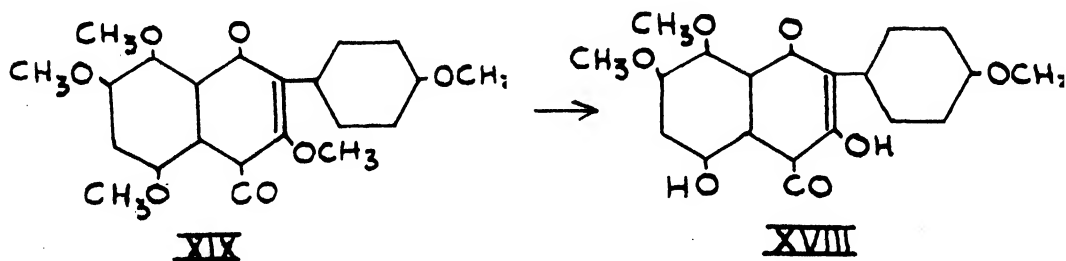
compound was stable to aerial oxidation in alkaline solution. This last evidence did not appear to be quite dependable. For we had found in the course of other work that compounds like galangin and kaempferol

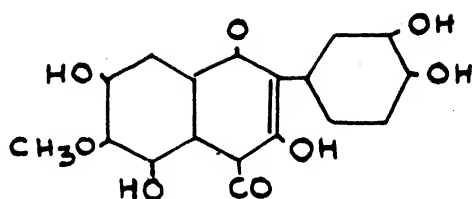
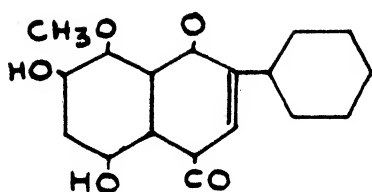


having a free hydroxyl in the 3- position and a small number of hydroxyl groups in the other parts of the molecule are fairly stable when exposed to air in alkaline solution (milder condition), and Perkin<sup>12</sup> had recorded that when air is aspirated through an alkaline solution of galangin-3-methyl ether (more drastic condition), it is oxidized with the formation of benzoic acid and phloroglucinol. The method cannot, therefore, be considered to be conclusive for the present purpose. In view of the above-mentioned synthetic work, the third methoxyl group should be located elsewhere. Of the two possible positions, 5 and 7, the former is known to be resistant to methylation and is the last to be affected in the plant or in the laboratory. Consequently, position 7 seemed to be more probable. Further, the constitution of 7:8:4'-*o*-trimethyl herbacetin (XVIII) seemed to be in accord with the lower melting point of tambulin. This could be checked by synthesis. A substance of this constitution could be most conveniently made by the partial demethylation of herbacetin-pentamethyl ether (XIX) using anhydrous alumi-

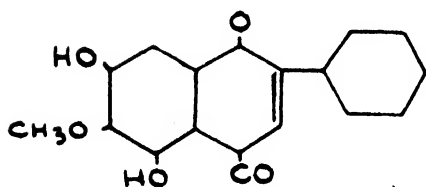
nium chloride in nitrobenzene solution. This method of simultaneous attack of the 3- and 5- methoxyl groups had been adopted successfully in a number of analogous cases and the synthesis of partial methyl ethers of galangin, kaempferol and quercetin reported earlier<sup>13</sup>. The reaction was found to proceed smoothly in the present case<sup>14</sup> and the product was found to have the properties reported for tambulin; a similar agreement was found between the acetates. Hence the constitution of tambulin should be considered to be that of the 7:8:4'-*o*-trimethyl ether of herbacetin (XVIII).

In conclusion a word is necessary regarding the structural interest in the colouring matter of *tambul* seeds. In tambuletin and tambulin, the hydroxyl in the 8- position is found to have undergone methylation in preference to those of the 7- and 3- positions, though biogenetically there is clear indication that the former should have a later origin than the latter. The reasons for this preferential partial methylation and its mechanism are not yet known. Under this category comes the flavone methyl ether wogonin

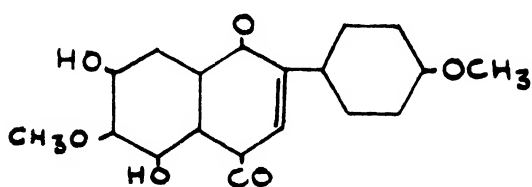




XXI



XXII



XXIII

(XX) which is found in the roots of *Scutellaria baicalensis*. A parallel series is presented by the 6-methyl ethers like patuletin (XXI), oroxylin-A (XXII) and pectolinarigenin (XXIII) in which the 6-position is preferentially methylated.

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# REVIEWS

**A Source Book in Greek Science**, by Morris R. Cohen ( McGraw-Hill Book Co. Inc., New York ) 1948, pp. xvi+579.

THE PRESENT BOOK IS THE FIFTH IN THE series of source books in the history of the sciences published under the general editorship of Prof. Gregory D. Walcott. The earlier volumes in the series dealt with astronomy, mathematics, physics and geology respectively. A source book in medieval science is also to be issued, and, further, a volume containing the most important contributions of the major sciences from 1900 to 1950 is planned for publication by about 1960 ; and, according to the general editor, a similar volume is to be issued each half century thereafter.

Stimulated largely by the growing concern in recent years of scientists by the impact of science on society, the history of science and its social implications has attracted considerable attention. Of all the adventures of the human mind, the scientific adventure has proved the most fruitful and profound. In fact, the very idea of " progress " is comparatively modern : it is a result of the scientific and technological revolution. As W. K. Clifford, noted for his " profound and orderly understanding ", said in his book *The Commonsense of the Exact Sciences* published in the last century : " scientific thought is not an accompaniment or condition of human progress, but human progress itself ". In our own day, Sarton, the distinguished historian of science, has emphasized in his *Study of the History of Science* that " the acquisition and systematization of positive knowledge are the only activities which are truly cumulative and progressive. The history of science is the only history which can illustrate the progress of mankind. " With the increasing specialization that is inevitable with the development of science and with its growing utilization by society, it has become all the more necessary for scientists, and no less for others, to acquire a proper understanding of the limitations of science and of the factors that determine its progress and development. This understanding, and also a balanced science education, is greatly

helped by a study of the history of the growth of science under different civilizations and social environments and how it has acted and reacted on society. The President of the Harvard University, Dr. J. B. Conant, has vigorously advocated in his highly interesting book, *On Understanding Science*, the historical approach in science teaching. Dr. Conant has urged that the strategy of science can best be understood by a study of typical case-histories and he has provided most illuminating illustrations of them in his book, taken mostly from 17th and 18th century science.

The publication of the present source book in Greek science is most opportune and the work is assured of a wide welcome. As the author points out in the preface, the extracts from Greek science presented in the book cannot take the place of a history of science, nor can they offer a systematic study of Greek science or of the position it occupied in Greek civilization. " But no history of Greek Science can convey an adequate picture, unless the readers see something of the original source on which the historian relies. The presentation of these sources seems all the more desirable, because the material on which we draw is often fragmentary, and scattered in many diverse volumes. " The study of the volume will help in correcting, what the author calls, an established popular error, namely that the Greeks were merely speculators and that the natural science began in the 17th century with Galileo and his successors.

The book opens with a section on mathematics, and it is in mathematics, particularly in geometry, more than in any other branch of science that the Greeks made their greatest contribution. We find here the proof ( Euclid ), oft-repeated for the last two thousand years, that  $\sqrt{2}$  is an irrational number. We read here ( page 20 ) the well-known theorem that the number of primes is infinite. An interesting extract from Archimedes is on the sand-reckoner. The sand-reckoner described a system of representing numbers different from the usual Greek system, and it allowed representation of any number, however large. To illustrate the utility of the system, Archimedes estimated the



number larger than the number of grains of sand necessary to fill the universe. (Curiously enough, the estimate of the Syracuse philosopher-scientist is comparable to the number of fundamental particles in the universe estimated more than 2,000 years later by Eddington.)

Extracts are given from Aristarchus of Samos (the Copernicus of antiquity) on the sizes and distances of the sun and moon. Also extracts are given dealing with the precession of the equinoxes. Ptolemy's arguments for a fixed earth are reproduced from his *Almagest*. The section of mathematics and astronomy covers a little less than 200 pages.

Next comes the section of physics of about the same length. We find here extracts from Aristotle on the equations of Aristotelian dynamics. Aristotle maintained, among other things, that "within bodies moving with their proper motions, the larger moves quicker". Again, we have an extract from Philoponus (page 279) contending that Aristotle was wrong in his assumption that a larger weight falls in a given time through a greater distance than a smaller weight does in the same time. The Greek mechanics, like their astronomy and a good deal of physics, was based not on experimental observation but on deductions drawn from general principles—the general principles being assumed on philosophical and speculative rather than on scientific grounds. Witness, for instance, Aristotle's dictum that nature abhors vacuum.

Reasoning by analogy (for instance the analogy between macrocosmos and microcosmos) played a significant part in the derivation of principles. This gave them a wide sweep, but as the analogies were often superficial (in the scientific sense), the principles so derived were of little value in natural science.

Interesting extracts are given relating to siphon, simple machines, and so on. The last 150 pages of the book are devoted to zoology, botany, animal and human psychology. Here we have interesting extracts taken from Aristotle who, for his acumen as a biologist, ranks supreme in the same sense that Archimedes does in the realm of mathematics and physics. Here one finds reference to Indian elephants (page 537). Interesting extracts from Aristotle relate to the senses of animals: "There is no doubt but that fishes have the sense of taste. . . With regard to sight and hearing

we cannot make statements with thorough confidence or on irrefutable evidence."

It is obviously difficult to summarize the contents of a work of this kind, and more so for a reviewer who can claim no expert knowledge of this fascinating subject of Greek science. The book covers an extensive field and it appears that practically every work of interest and importance finds a reference in it. It is a book that should find a place in every library concerned with science and its teaching.

D. S. K.

**The Chemistry and Manufacture of Indian Dairy Products**, by K. S. Rangappa & K. T. Achaya (Bangalore Printing & Publishing Co. Ltd., Bangalore City), 1948, pp. 189. Price Rs. 10.

THE IMPORTANCE OF DAIRY INDUSTRY HAS not been adequately recognized in India and it is, therefore, an encouraging sign when a book on the chemistry and manufacture of Indian dairy products appears in the market. As has been correctly pointed out by Professor J. C. Ghosh in his foreword to this book, the Indian dairy products "are fundamentally different in nature from the dairy products in Europe and America" and hence the problems connected with these products need considerable research and extensive application of the results of research before the dairy industry can be built up on a firm scientific foundation. Unfortunately, however, research in dairy industry, not being as glamorous as that in many other subjects, has not been financed or encouraged as much as its importance would deem it necessary. What little has been achieved by a few workers in Government laboratories or research institutions needs to be extended and more encouragement by way of better-equipped laboratories, conditions of employment, etc., is needed before the results of our research investigations can be usefully employed by the industry. It would, however, be unsafe to incorporate into a text-book results which have not been tried out under a variety of conditions or confirmed by a few other workers. For example, on page 72, quoting the work of Roy and Bhatnagar, the authors of the book point out that during fermentation of milk its nitrogen content increases. To the best knowledge of this reviewer, this observation of nitrogen fixation in milk

has not been recorded by any other worker in India, Europe or America. It would have been interesting to read some hypothesis postulated to explain this rather unusual phenomenon.

The book has been divided into 3 parts : part I—milk and unfermented milk products ; part II—fermented milk products ; and part III—ghee. Since this book deals primarily with dairy products peculiar to India, products such as ice-cream and milk powder, which are also being manufactured and used in India to an increasing degree in recent years, should have been included.

Discussing the bacterial quality of milk marketed, the authors quote reference (page 51) to a book published in 1916 to point out that "it is encouraging that the samples were free from pathogenic organisms like *B. Typhus*, *tuberculosis*, *diphtheria* or *cholera*". This is most misleading, since it is well known that the detection of pathogenic organisms in milk is quite a difficult and laborious procedure. Incidentally, the sentence does not read well in a book, especially, since the name of the first is now recognized as *Eberthella typhosa* and the latter 3 are names of diseases and not organisms. The authors emphasize that under Indian conditions it is quite easy to produce milk of bacterial count of 30,000 or less per ml. (page 49). Actually, in the results reported in Table XXX, even in the herd milk at the Government farm of *Indian Dairy Research Institute*, the bacterial count was about 100,000 per ml. It will need a considerable reorganization of our dairy methods before milk of a bacterial count of the order of 30,000 per ml. can be produced. Again, on page 55, it is pointed out that "And it is to this practice (of heat treatment of milk) that the freedom from milk borne diseases in India is to be mainly attributed". It is well known that in many homes the milk is not heated to a sufficiently high temperature or for sufficiently long periods to ensure the complete destruction of pathogenic organisms. If we do not hear of diseases transmitted through milk, this reviewer believes that it is largely due to lack of adequate public health surveys and "follow ups" of epidemics rather than the absence of such milk-borne epidemics.

The arrangement of the textual matter is good, although many printer's mistakes

need corrections. To cite a few glaring examples : page 140, line 15, "interreaction ship" instead of "inter-relationship" ; page 141, "voriations" instead of "variation", etc. The get-up of the book is excellent although many research workers in the field may find the price of Rs. 10 somewhat beyond their means.

1.

**Turning and Boring Practice & Drilling and Surfacing Practice**, by Fred H. Colvin & Frank A. Stanley (McGraw-Hill Book Co. Inc., New York, 1948, pp. xv+531. Price \$4.75.

ALTHOUGH THE AUTHORS HAVE WRITTEN two books separately, one on *Turning and Boring Practice* and the other on *Drilling and Surfacing Practice*, in the opinion of the reviewer these are considered as two volumes under general machine-shop practice. A comprehensive survey of modern machine-shop practice becomes increasingly difficult because of the constant changes in machines, methods and materials employed. For example, where formerly the planer and shaper machines handled all flat-surface work, the milling machine, grinder and more recently the broaching machine are now used almost exclusively on production work. In addition to new machines, new methods have come into practice with the introduction of new materials.

The authors have made these points quite clear in these two volumes and, in their opinion, all those connected with a machine shop, be he an executive or a workman, should have wider knowledge of factors which affect the smooth working of a machine shop. The authors have also emphasized the fact that principles of machine operation have remained unchanged even though vital changes in the use of machines have come to stay in a modern machine shop.

In the first volume of the work, the authors concern themselves with turning and boring practices. This volume is divided into five sections. The chapters on turret and semi-automatic lathes are written very exhaustively and very few books give so many details about the new techniques employed in production work. Boring has received special attention in a separate section. A large number of illustrations given in this section showing how to execute large boring operations will be found very useful.

The section on cutting tools for different materials contains valuable information such as use of carbide tools, suggestion and methods for machining various metal parts, cutting oils and other coolants. Also described in the same section are the methods to be employed in case of non-metallic materials such as rubber and plastics.

As drills and drilling machines are used to a greater extent than other machines in machine-shop practice, it is quite appropriate that the authors have devoted a complete volume for "drilling and surfacing practice". This volume runs into six sections describing in detail drilling, reaming and tapping, planers and shapers, milling, milling cutters and broaching.

These volumes are very authoritative covering almost all the fundamental machine-shop operations. A special feature of this work is the inclusion of a large number of tested, new workshop practices to make difficult jobs easier, and many short-cuts employed in the mass production are also elaborately discussed. Great stress is laid on the economy of time and money in all the operations.

The authors need no introduction and their work is so thorough and exhaustive that the reviewer strongly feels that these books will prove to be valuable additions to all technical libraries and that all those connected with machine-shop industry could use them with great advantage.

**Handbook of Indo-Australian Trade — 1947**, compiled by E. V. S. Maniam & M. L. Gupta (Bureau of Economic Research, Kanpur), 1948, pp. x+60. Price Rs. 5.

THIS PUBLICATION WHICH IS THE RESULT of an economic survey of the two countries, is a sequel to the widespread desire felt in both the countries for increased trade relationship. It presents in a handy form valuable information regarding the possibilities of Indo-Australian trade.

It may be recalled that an Indian Industrialists' Delegation visited Australia in 1945 and a similar Delegation from that country came to India in 1946 to explore ways and means of fostering trade between the two countries. The Bureau of Economic Research, Kanpur, was asked to prepare a factual memorandum for the guidance and information of the Australian Delegation, and the present volume is an enlargement on the report.

The handbook gives, in a concise form, facts and figures which are not easily available elsewhere. It is an exhaustive survey of the economic resources of the two countries and indicates the deficiencies of each country which can be met by the other. The different offices under the Commonwealth Ministry, Diplomatic and Trade Consulates and others directly and indirectly concerned with the administration of trade, commerce and industry have been mentioned to facilitate trade inquiries. The names and addresses of the different industrial and commercial bodies in Australia have also been given and these will be very helpful to Indian exporters.

The handbook also contains a directory section, giving names and addresses of Australian and Indian manufacturers and import and export houses in the two countries. The directory is classified under different heads and alphabetically arranged for quick reference.

**The Manufacture of Iron and Steel, Vol. I—Iron Production**, by G. Reginald Bashforth (Chapman & Hall Ltd., London), 1948, pp. viii+228. Price 21s. net.

THE AUTHOR HAS NEATLY COVERED IN FAIR detail practically all the aspects of iron production. It should prove extremely useful to students of metallurgy. A typical feature of the book is the list of references relating to the voluminous data condensed in the text.

The contents are well laid out and balanced. Reference to Bihar and C.P. haematite iron ore belts has not been made in relation to India's iron ore deposits, although Mysore magnetite deposits have been referred to. It may be pointed out that the Bihar and Bengal iron ores form one of the richest iron ore deposits of the world. Further chapters deal effectively with the technical processes involved in iron and ferro-alloys' production.

The language of the book is precise and the information contained should prove useful both to students as well as to industrialists for reference and study. The technical details of the processes are dealt with comprehensively. The pages on electric reduction of pig iron, however, lack adequate reference to the Swedish practice. This may be ascribed perhaps to lack of space.

The book is a very useful addition to textbooks on iron production.

B. R. NIJHAWAN

# Developments in Vegetable Oil Extraction

M. N. KRISHNAMURTI

*Volkart Brothers, Bombay*

**D**URING the past 25 years, the vegetable oil extraction industry has undergone a considerable change with respect to processes, equipment and practices. As a result, the industry has now changed over from pressure extraction with expellers or hydraulic presses giving a comparatively low yield of oil, to processes based on advanced knowledge of chemical engineering principles yielding higher percentages of oil. This transition has been, for the most part, from an industry requiring a very modest capital investment in simple equipment to an industry representing a substantial investment in complicated chemical engineering equipment and auxiliary processes. Consequently, this higher yield of oil obtained from seeds or pressed cakes leaves the meal with a higher percentage of protein concentrate which provides higher nutritional value from the point of view of cattle feed. Moreover, the meal produced can be used with complete satisfaction for human consumption.

Soya-bean or, for that matter, any oil seed with an original oil content of less than 30 per cent is seldom pressed by mechanical expellers in America or on the Continent. For seeds with an oil content exceeding 30 per cent, such as groundnut, a combination of expeller-solvent extraction operation is recommended. Most of the earlier installations in Europe and the United States adopted this process for extracting oil from soya-beans, for this was a comparatively low oil-bearing seed with 20-22 per cent oil content, and mechanical extraction was expensive, as the oil content in the residual cakes could not be reduced to less than 4.5-5 per cent. The solvent extraction process, on the other hand, yielded a cake containing only 0.5-1 per cent oil. Later installations have demonstrated the benefits of solvent extraction of oils from pressed cakes obtained by expellers from groundnut which contain 8-10 per cent of residual oil, and there were several mills operating in Germany employing expeller cum solvent extraction processes before the war.

## Process

The raw materials, whether they be nuts, beans or pressed cakes, with an oil content not exceeding 30 per cent, are treated in the preparatory milling equipment, converting them into flakes of suitable size and thickness with moisture regulated to 12 per cent and temperature lower than the lower boiling limit of the solvent. The flaked material then enters the extractors where it contacts the solvent, and oil is removed by washing and diffusion. The mixture of oil and solvent termed "miscella" is clarified by filtration and, subsequently, by centrifuging. The mixture is then distilled to remove the major part of the solvent, and finally stripped in rectifying columns to remove the last traces of moisture and solvent. The solvent-free oil is drawn off the base of the stripping column and pumped to storage tanks. The solvent vapour from the evaporator and distilling columns is condensed and water removed by decantation before returning the solvent to the extractor. The solid residue from the bottom of the extractor enters dryers where the last traces of solvent are driven off by heat and condensed. The solvent-free meal is conditioned by toasting followed by cooling, sifting and bagging in the warehouse.

## Equipment

A solvent extraction plant consists mainly of the extraction apparatus, evaporator units, distilling and rectifying columns, "miscella" storage tanks, filters, meal dryers and accessories. The Anderson extraction plant discussed here is of American design. It has an extraction column embodying the patented settling chamber device for "miscella" fines and a discharge device which is a modification of Anderson's expeller application. The settling chamber device consists of a number of vertical tubes through which the "miscella" flows. A disc in which there is a triangular opening covers the top of the tube permitting the intermittent discharge of "miscella" from each tube as the disc revolves. While the "miscella"

in each tube is stationary, the fines settle into the lower part passing out through the bottom of the column with the flakes of the material.

One of the latest models of the continuous solvent extraction plant is now operating near Wooster, Ohio, U.S.A. The plant does not require any elaborate factory installation. It is placed in the open with no building of any sort, thus eliminating explosion hazard inherent in housed equipment, especially when hexane is used as a solvent. The extraction unit is located 50 ft. away from the preparation building. It is thoroughly insulated and weather-proofed. The equipment is largely pre-assembled and compact, different pieces of the unit supporting one another so that no special building or frame-work is needed except for a concrete footing and foundation slab.

#### Remote Control

The outdoor extraction unit is remotely controlled from a room in the preparation department by one operator. Push-button station, complete with pilot lights for all equipment, are grouped in accordance with the operating sequence. All principal equipment of the extraction unit are electrically interlocked. It is, therefore, necessary to start the plant in proper sequence and interruption to any unit shuts down equipment ahead of it. The grouping of all instruments at one location gives the operator very close control and allows finer co-ordination of the entire operation.

The vapour scrubbing device located between the main condensers and the solvent recovery dryers afford protection to the main condenser by separating dust that might come off with the vapours from the solvent recovery dryers which, if allowed to enter the condenser unit, affect the efficiency of the condensers, thus increasing the solvent loss. The equipment is designed with two main condensers, each having a sufficient capacity to take care of the entire plant in case any cleaning operation should ever be necessary while the extraction unit is in operation. The evaporator, vaporizing chamber and stripping column are built in one column with the further advantage of using waste heat from one element as the heat source for the other saving in steam, piping and insulation. This solvent extraction unit is elegant in design, practically trouble-free, and requires less labour and space.

#### Solvents

The selection of the most suitable solvent depends on the following considerations:

The solvent desirable for vegetable oil extraction should take up the oil selectively and dissolve a minimum quantity of wax, resin and colour; otherwise this may result in a dark crude oil involving high refining costs. The solvent should be low boiling with a narrow boiling range and chemically inert, leaving no odorous residue. Among the solvents now in use are alcohol, carbon-disulphide, chloroform, ethyl ether, methyl chloride, acetone, benzene and petroleum ether. In Europe, the common solvent recommended by the manufacturers of equipment is benzene, b.p. 70°-90°C. This has an aromatic flavour and, in order that no aromatic smell is present in the products, the meal is de-aerated in the process. In the United States, where the continuous system is followed, the solvent recommended is normal hexane, b.p. 62°-65°C. Both the solvents have good selectivity, excellent stability and low evaporation residue, but they are inflammable. The Japanese have reported the use of alcohol as a solvent for the batch extraction of soya-beans in a plant at Darien, Manchuria. They have published data on the miscibility of soya-bean oil in various ethanol water ratios. The data shows that absolute alcohol (99.52 per cent) is miscible in all proportions with soya-bean oil above 67.3°C. At the lower alcohol concentration, 95.92 per cent, the oil solubility is greatly reduced, and complete miscibility is not attained even at the boiling point, 78°C. However, the solvent proportion of 95 per cent alcohol may be brought into a practical solvent range by operating under sufficient pressure to bring the temperature to 90°C.; obviously, the separation of the miscella from the oil-bearing material must also be carried out under pressure. Both 95 per cent alcohol and absolute alcohol remove water from oil-bearing meal at the moisture content usually employed in solvent extraction, and quickly becomes diluted to such an extent as to be of no further use as an oil solvent unless rectification and dehydration be used to remove the water. To minimize the dilution of alcohol, the Japanese recommended that the moisture content of the oil-bearing material be reduced. In fact, under the ordinary conditions of drying, the moisture content of soya-beans is often reduced below 3 per cent and an actual improvement

takes place in the efficiency of alcohol as an oil solvent.

### Solvent Loss in Tropical Climates

Solvent loss is the chief consideration in all solvent extraction units. Various equipment manufacturers have adopted variations in design in order to see that the solvent loss is kept within a reasonable limit. In all the designs the local considerations as to air temperatures, humidity and temperature of cooling water available, physical condition of raw material while entering the extractors, have been given careful consideration. The average summer temperature of cooling water available in this country is about 90°F., while the temperature of water on the Continent and in the United States is only 65°F. To cool the temperature of water from 90°F. to 65°F. requires a big refrigeration unit, which means the process is uneconomical. For this the condenser should be designed for a high flow rate of cooling water; if the solvent loss turns out to be high, the vent gases can be directly led into a dephlegmator unit where it can be cooled by a small refrigeration unit and condensing the solvent vapour sufficiently well below the vapour dew point.

### Cost Estimates per 24-hour Day Basis for Solvent Extraction of Groundnut Pressed Cakes

#### Raw Materials —

	Rs. a. p.
50 tons oil cake of approx. 10% oil content at Rs. 130/- per ton	6,500 0 0

	Rs. a. p.
<i>Utilities —</i>	
(a) 70,000 lb. process steam at 140 lb. per sq. in. corresponding to 4.6 tons coal at Rs. 30/- per ton	138 0 0
(b) 13,750 gal. make-up water requirement, i.e. 5% of the total water circulated at Re. 1/- per 1,000 gal.	14 0 0
(c) 1,500 kWh. electricity at ½ anna per unit	47 0 0
(d) Solvent hexane based at 1% loss equivalent to 120 gal. per day (Price Rs. 3/- per gal.)	360 0 0
	185 0 0
<i>Labour &amp; Supervision —</i>	
1 Technical manager -- Rs. 900/- per month	
3 Solvent plant operators -- Rs. 1050/- per month	
3 Assistant solvent plant operators -- Rs. 600/- per month	
3 Mechanical engineers -- Rs. 900/- per month	
3 Asst. mech. engineers -- Rs. 450/- per month	
3 Boiler engineers -- Rs. 600/- per month	
6 Preparatory plant tenders -- Rs. 600/- per month	
Depreciation at 10% on Rs. 10,00,000 per year	335 0 0
<b>TOTAL PER DAY</b>	<b>7,579 0 0</b>
<i>Credit for Sale Proceeds</i>	
4 tons of oil based at 8% recovery and selling at Rs. 1,125/- per ton	4,500 0 0
46 tons oil cake at Rs. 100/- per ton	4,600 0 0
<b>TOTAL</b>	<b>9,100 0 0</b>
Profit per day Rs. 1,521/- (45% over investment)	

#### REFERENCE

1. BEEKEL, A. C. : *J. Amer. Oil Chemists Soc.*, 1948, **25**, 7.

## Non-Technical Notes

### RUBBER-LINED EQUIPMENT FOR CHEMICAL INDUSTRIES

RUBBER OFFERS A HIGH DEGREE OF RESISTANCE to many corrosive liquids and, therefore, rubber-lined equipment is finding increased applications in the processing of corrosive substances in chemical industry. Rubber can be made to adhere strongly to materials like iron, steel, wood and concrete.

The usual practice followed in industry for the rubber-lining of equipment is to make suitably prepared sheet rubber adhere to the basic material by the use of adhesives. Rubber latex has been used only on a limited scale in rubber-lining.

Methods for using latex for the rubber-lining of equipment have been developed. The standard procedures have been modified and adapted to suit Indian conditions.

Actual working details of the processes are covered by Indian patents, and the "know how" will be made available to parties taking up commercial production according to these patents."

### Process

The methods employed for rubber-lining are :

- (i) electro-deposition of rubber ; and
- (ii) ionic deposition of rubber.

(i) *Electro-deposition*—This method is particularly suited for small metallic equipment of complicated shape. The article to be lined is first subjected to a pre-treatment. It is then immersed in a properly compounded latex bath from which rubber is anodically deposited on the article.

Methods have been developed to improve the mode of preparation of the latex bath, and also to improve the quality of the rubber-lining obtained.

(ii) *Ionic Deposition*—This method can be used for rubber-lining chemical equipment of large size.

Films of suitably compounded latex are made to adhere to pre-treated surfaces of articles employing the principle of ionic deposition. A wide range of rubber compositions to suit the requirements of individual industry has been developed.

In addition to the general application of this method, special mention may be made of the fact that wooden boxes can be rendered suitable for use as storage battery containers by lining with rubber.

### Raw Materials

The raw materials and chemicals required for these processes are easily available in India.

### Equipment

The process can be advantageously exploited by existing rubber-goods manufacturers. Most of the equipment required would be available with them. For general guidance, a list of equipment needed is given below.

*Approx. cost*

	Rs.
(1) One porcelain ball mill (without motor ; approx. capacity, 2 gal.)	800
(2) One mixer (without motor ; to be fabricated locally ; approx. capacity 2 gal.)	300
(3) One vulcanizer ( to be constructed locally )	1,000
(4) Three motors	1,200
(5) Three rubber-lined, mild-steel tanks ; (i) zinc plating, (ii) rubber deposition, (iii) washing, each 3'×3'×4', @ Rs. 200 each ( rubber-lining may be done locally )	600
(6) One air compressor ( with motor and shaft system )	2,500
(7) One spray gun ( medium size )	200
(8) Building with 2 rooms, 13'×15' each	5,000
<b>TOTAL</b>	<b>11,600</b>

### Cost Sheet

*Electro-deposition Basis —*

Manufacture of 50 sq. ft. rubber-lined area per day of 8 hours :

*Working Capital —*

	Rs.
1. Power at 1 anna per unit, 400 kW. for zinc plating and 1,425 kW. for rubber-lining of 50 sq. ft.	114.06
2. Rubber chemicals including pure zinc anodes and water	20.00
3. Depreciation	1.00
4. <i>Management &amp; Labour —</i> 1 Assistant chemist at Rs. 250/- p.m. ( 24 working days ) 4 Coolies at Rs. 90/- p.m. each ( 24 working days )	25.4
5. Miscellaneous	5.00
<b>TOTAL</b>	<b>165.46</b>

Estimated cost to rubber-line 1 sq. ft. area : Rs. 3/4/0.

*Spraying Basis —*

Manufacture of 100 sq. ft. rubber-lined area per day of 8 hours :

	Rs.
1. Power at 1 anna per unit ( 1,200 kW. )	75.00
2. Chemicals including water	40.00
3. Depreciation	1.00
4. <i>Management &amp; Labour —</i> 1 Chief chemist at Rs. 350/- p.m. 6 Coolies at Rs. 90/- p.m. each 1 Steno-typist, at Rs. 150/- p.m. ( 24 working days )	43.00
5. Miscellaneous	3.0
<b>TOTAL</b>	<b>162.00</b>

# NOTES & NEWS

## Mold Amylase by Submerged Fermentation

THE MOLD-BRAN METHOD AND the amylo method are two procedures normally employed in commercial practice for the production of mold enzymes. From the standpoint of distillery operation, however, both these methods possess certain disadvantages. In mold-bran production, considerable labour, processing space and equipment, as well as a cereal bran substrate are required, while in the amylo process the entire mash volume must be pre-liquefied and aerated under pure-culture conditions. As a result of a survey conducted at the Northern Regional Research Laboratory, Peoria, U.S.A., a few molds have been isolated which are capable of producing starch-hydrolysing enzymes when cultured under submerged conditions (*Ind. Eng. Chem.*, 1949, **41**, 100). Laboratory studies showed that culture liquor from one strain, *Aspergillus niger* NRRL 337, completely replaced malt when added to corn mash at the rate of 10 to 20 per cent of the final mash volume.

Pilot-plant experiments were conducted to obtain data on the following points: extent of enzyme production in deep tank fermentations under varying rates of aeration; the relationship between enzymatic potency of the mold culture liquor and the quantity required to replace malt completely; alcohol yields obtainable by the continuous use of fungal amylases for the saccharification and fermentation of corn; and design and cost of operation of submerged process for production of fungal enzymes.

The results showed that saccharification was satisfactory and alcohol yields were comparable to those obtained with malt when mold culture liquor equivalent to 6 to 10 per cent of the final mash volume was used. The cost of amylase produced by this process is estimated at 6.06 cents per bushel of grain processed, as compared with 12.1 cents in the case of malt.

Corn meal, starch, molasses, maltose and commercial glucose were found to be suitable sub-

strates. Corn-steep liquor, stillage, soya-bean meal and animal tankage were found to be good sources of protein. Calcium carbonate appeared to be beneficial in maintaining optimum pH levels during fermentation. Sterile air is supplied at a rate of 0.25 vol. per vol. of medium per min. Mechanical agitation was also employed. Malt stillage can be replaced satisfactorily with mold stillage as the substrate for mold cultivation and can be used continuously.

## Concentration of Latex

A PROCESS FOR THE RAPID CONCENTRATION of Type III GR-S latex is described (*Ind. Eng. Chem.*, 1949, **41**, 156). The process, which is an improvement on the German method of latex concentration, permits a more rapid concentration of GR-S latices to solids content considerably higher than were achieved by the German method. The process involves gelation by the addition of an electrolyte (sodium chloride) and cooling, and subsequent processing of the mix to yield a fluid and rapidly filterable mass.

The sample of GR-S latex to be concentrated is analysed for soap and rubber content, and if the soap content is less than 0.25 milliequivalent per gm. of rubber, a solution of potassium soap of K. wood rosin is added to bring the soap content to 0.28 to 0.29 milliequivalents. The pH is maintained between 10.5 to 11.5 by the addition of requisite quantity of sodium hydroxide solution. In the next stage 3N. sodium chloride is slowly added to the latex to give a final concentration of approximately 0.24N. sodium chloride in the final volume of latex and salt solution. It is essential that all the additions to the latex are made with agitation. The salt latex-salt mix is cooled as soon as possible after preparation by surrounding the vessel with an ice-bath. During cooling the mass should be agitated continuously. The cooling is continued through the gelled stage and the subsequent thinned stage until the temperature of 5°-8°C. is reached. The material is now ready for filtration

through a double-walled Buchner funnel, pre-cooled by ice-water. It is preferable to over-concentrate the latex and dilute it back to the required concentration. The filter cake is transferred to a closed container, allowed to warm up to room temperature and blended by agitation.

Exceptionally stable latices of 60 to 70 per cent solid contents and above can be obtained by this method, and they exhibit all the characteristics of a good latex. Pilot-plant studies have shown that the process works well in large-scale practice, and suitably prepared Type III GR-S latex can be filtered readily on an Oliver filter.

## Physical Constants of Leather & Collagen

INVESTIGATION CONDUCTED AT the National Bureau of Standards, U.S.A., on leather has added fundamental data on the physical and physico-chemical properties of leather and collagen (*J. Franklin Inst.*, 1949, **247**, 155). A knowledge of these constants may permit utilization of leather in applications requiring specific properties possessed by leather but as yet unknown. The effect of tannage and other treatment on values of such constants may yield valuable information concerning the structure of collagen and the mechanism of tanning.

Since leather is a porous material containing as much as 60 per cent void space, most measurements of physical properties of the leather substance present obvious difficulties. One method of solving this problem lies in completely filling the leather with a liquid and ascertaining the properties of the liquid-leather system. This approach has been used at the Bureau's leather laboratory to obtain data on the cubical expansivity of leather. Using water as the confining liquid, dilatometric measurements of a water-leather system were made and compared with measurements on a similar system containing water alone. As a result of these measurements it was found that the average expansivity of  $540 \times 10^{-6}/^{\circ}\text{C.}$  applied for collagen and all leather except chrome-vegetable leather. The average expansivity of the latter tannage is  $340 \times 10^{-6}/^{\circ}\text{C.}$  These averages have reproducibilities as indicated by standard deviations of  $13 \times 10^{-6}/^{\circ}\text{C.}$  and  $37 \times 10^{-6}/^{\circ}\text{C.}$ , respectively.



Leather, when heated in water above a well-defined temperature, depending on tannage and other factors, undergoes shrinkage. The temperature at which shrinkage begins is defined as the shrinkage temperature and is considered a measure of the efficiency of the tanning process—the higher the temperature, the more thorough the tannage. During the dilatometric studies it became evident that the apparent shrinkage actually represented an increase in real volume. This increase in volume amounted to approximately 1 per cent and was irreversible in character although the purely thermal expansion was found to be nearly completely reversible. It was also observed that this increase in real volume occurred very slowly at temperatures well below the shrinkage temperatures of the specimens found by regular test methods. This indication that shrinkage was a rate process was substantiated by a rate measurement which indicated that the expansion occurring during shrinkage follows a first order process.

### Improved Guayule Rubber

THE IMPROVEMENT OF GUAYULE rubber by shrub retting has been recently investigated (*Ind. Eng. Chem.*, 1949, 41, 346). By retting, the resinous contaminations of the crude rubber are reduced to about half the quantity present in unretted guayule rubber, tensile strength is increased by 50 per cent and some other physical properties are improved.

For rapid retting, shrub moisture content is to be maintained between 35 to 55 per cent on a wet weight basis. Temperatures should never exceed 60°C.; for unmixed or occasionally mixed shrub, the safe limit is 55°C.; for continuously mixed shrub, temperature should be maintained from 25°C. to 35°C. All degrees of mechanical reduction from whole shrub to finely crushed, shrub may be used. More finely cut or crushed shrub will ret more rapidly, but is suitable only for mixed rets, while coarsely cut or whole shrub may be used only for unmixed rets. The natural pH of the shrub is satisfactory for initiating the retting process.

The methods of retting, which have been investigated, fall into 2 categories: those not involving any disturbance of the shrub during the retting period, and

those involving occasional or continuous mixing. In the first category, floor retting and bale retting gave satisfactory retting. Depth of piling must be correlated with the degree of mechanical reduction and the retting time will be from 1 to 2 weeks. Whole shrub can be retted in the bale following parboiling, defoliation, and rebaling, with a retting period of approximately 2 weeks. In the second category are included floor retting with occasional mixing, and drum retting with continuous mixing.

Retting on the floor with periodic mixing (4 to 6 times a day) requires that the shrub be rather finely cut (0.5" followed by crushing), the depth of piling be closely correlated with the size of cut, and the retting period be adjusted to 6 or 7 days. Shrubs cut to 0.5" on a rotary cutter, piled 10" deep, and retted for 7 days has been found to give excellent improvement in quality and optimal recovery of rubber. Temperatures may range up to 60°C. or even slightly above in parts of the shrub, but the moisture content should be kept in the range of 45 to 50 per cent by adding water as needed. Drum retting provides the neatest and easily controllable method of retting. Aeration, moisture, and temperature can be kept under continuous control at the desired level, and very finely reduced shrub can be used without the danger of anaerobiosis. Continuous and uniform maintenance of optimal conditions permits a reduction of the retting period to 3 days.

### Plating of Plastics

A PROCESS, SIMILAR TO ELECTRO-deposition of rubber films from aqueous latex, has recently been developed at the Columbia University for the production of plastic-coated products (*Ind. Eng. Chem.*, Feb. 1949, 15A). Sheets of uniform thickness are obtained and complex "hollow" shapes can be "plated" from a plastic emulsion and can be conducted in ordinary plating tanks. However, this technique produces a film less than a millimeter thick and building up of thicker films is time-consuming and does not produce a uniform sheet.

The latices used may be prepared from any plastic material which will form a stable aqueous emulsion containing 55 per cent solids. A plating tank divided

by a thin barrier of porous porcelain is used. The latex is placed in the anode compartment of the plating tank, the cathode chamber being filled with a 50 per cent sodium chloride solution. The porous divider is necessary to prevent hydrogen evolved at the cathode from migrating to the anode and interfering with the deposition of the plastic. Copper cathodes may be used since the cathode does not dissolve into the electrolyte.

The choice of the anode is important. Nickel, which has proved impracticable for the electro-deposition of rubber, is especially effective for plastics. Zinc, which is widely used for rubber deposition, destabilizes vinyl type of plastics. Passivated metals such as aluminium tend to form a spongy film.

Metal-free films can be formed by depositing the plastic on a porous barrier, or mould of alundum or porous porcelain. Mercury, used as an anode either behind the barrier or contained in the body of the mould, forms insoluble anode products which do not penetrate the barrier.

If the coating composition contains a plasticizer, the film is coherent and firm but does not attain its maximum tensile strength until it has been baked for half an hour at 80° to 105°C. Unplasticized latices produce soft, crumbly films but these can be cured at a temperature of about 150°C. for an hour to produce a fused, hard sheet.

The tensile strength of the cured films is superior to that of dipped or sprayed film and approaches that of moulded products. This property could be enhanced by using emulsifying agents in the latex which would either volatilize or combine with the polymer during heat treatment.

The power requirements for electrolysis are small. When depositing on metal anodes, 2 to 4 v. are required, about one-fifth to one-tenth the potential difference used in the electro-deposition of rubber, deposition efficiency is about 2,500 gm. per faraday and at rates as high as 100 kg. per kW./hr. Best efficiency is obtained at rates between 10 and 20 kg. per kW./hr. with current densities of about 20 ma. per sq. cm. Under these conditions a 0.125" film can be deposited in about 15 min. on metal electrodes. Porous forms reduce the rate of deposition by about 15 per cent.

## Optical Glasses

A NUMBER OF NEW OPTICAL glasses, their properties and applications have been described (*Nature*, 1949, 163, 412).

With the introduction of barium crown glass, many new types of photographic lens became possible. This glass greatly improved all lenses of the air-spaced type. In 1934 samples of unusual glasses with characteristics  $n_D$  in the region of 1.85 and  $\nu$  the reciprocal relative dispersion of 47.0, were in existence.

Lanthanum is very soluble in boric acid, and its contribution to higher refractivity without increase of dispersion is remarkable. The oxides of tantalum, thorium and tungsten are soluble in the lanthanum borate base glass in amount up to 35 per cent. These new borate glasses are very stable and hard. They are harder than flints, stable to the atmosphere, and amenable to optical shop practices of moulding, grinding and polishing.

Early in the development work it was found that the rare-element borate glasses were extremely corrosive to all known pot refractories and platinum was employed for the actual production of these glasses. This was justified on the basis that no platinum would be lost by contamination, and that the glass, once homogenized, could be poured in its entirety, free from striae, into a single slab or into cast shapes without striae, seed, bubbles or other defects usually attending a glass made in a refractory pot. The first of the new glasses to be made had a refractive index of 1.7445 and a reciprocal relative dispersion of 45.8. The glass was slightly yellow, but further work established the origin of the yellow colour, and finally glasses were produced as colourless and homogeneous as any other optical glass.

Since 1940 the types of these glasses in production have been extended, and at the present time the following seven types are being made:

EK	$n_D$	$\nu$
— 110	1.69680	56.2
— 210	1.73400	51.2
— 310	1.74500	46.4
— 320	1.74450	45.8
— 330	1.75510	47.2
— 450	1.80370	41.8
— 448	1.88040	41.1

All these glasses contain thorium, and in the case of folding

camera, in which the lens may rest for a long time in close proximity to film, the radioactivity of the thorium may be a disadvantage. A thorium-free equivalent of EK — 320 is available.

The most useful of these new glasses were flint glasses containing titanium oxide and fluorine in addition to silica. The best glasses lie in the region 45 per cent silica, 28 per cent titanium oxide, and 27 per cent sodium fluoride, ranging in optical properties from  $n_D$  1.65/ $\nu$  29 to  $n_D$  1.58/ $\nu$  36.6. The glasses can be moulded, are very resistant to tarnish, and are easily fabricated by the usual optical methods.

The fluosilicate flints are almost as useful to the optical designer as the high-index glasses, since they extend the possible difference between crown and flint refractive index and between crown and flint dispersion. With these glasses, three-element lenses have been designed to give even better performance than the usual four-element types. K. H. Sun of the Kodak Research Laboratories has successfully produced novel mixtures in some 20 quite different glass fields including fluo-borate, fluo-germanate and fluo-phosphate systems.

An interesting group of glasses are those containing no oxides and composed entirely of fluorides. These glasses show the characteristic low refractive index and extremely low dispersion previously available only in fluoride minerals. The refractive index in most of the glasses is approximately 1.38 to 1.39 and the  $\nu$ -value, 100. Moreover, the glasses are transparent to below 300 m $\mu$  in the ultra-violet and to 5  $\mu$  in the infra-red, so that they may be very useful in the making of instruments requiring optical transparency over a wide range of wavelength. Difficulties are met with in the production of these all-fluoride glasses, but it is possible that these difficulties may be overcome in the near future.

## Failure of Railway Materials

WITH INCREASING SPEEDS, heavier revolving masses and greater overall weights of main-line locomotives, the need for sound materials both in the track and rolling stock has become of paramount importance. Fatigue and other causes of

fracture in engineering materials and parts have been widely investigated in recent years, and the root causes are now fairly well understood. So far as railway track materials are concerned, transverse fissures in rails at one time were a source of frequent discontinuity in service, although such occurrences have now been greatly reduced by improved methods of manufacture. As regards rolling stock, the highly stressed portions of locomotives are susceptible to fracture arising from fatigue cracks usually originating in faulty design or due to indifferent machining and finishing of surfaces.

The failures of rails through transverse fissures were first observed in the United States, where, up to 1932, no fewer than 65,281 cases had been reported, representing an average of 6.14 failures per 100 miles of track per annum. The true transverse fissure starts in the railheads, developing from a tiny shatter crack or hair-line crack and gradually spreading upwards and outwards until the unaffected sectional area is so weakened that it suddenly fractures transversely under a passing wheel load. The origin and development of hair-line cracks in the sectional thickness of steel is believed to be due to intergranular weakness derived from factors associated mainly with steel-making, fabrication and heat-treatment of the product. Data contained in the technical literature is not unanimous in attributing this phenomenon to any well-defined set of conditions. It is, however, understood that steel-making conditions involving the solubility of gaseous elements like hydrogen, oxygen and nitrogen in steel, the conditions of deoxidation in final processes of steel-making, teeming conditions and, last but not the least, factors associated with the heat treatment and fabrication of the product particularly in its final rate of cooling, all play a part in bringing about a state of intergranular rupture in the finished product. Unfortunately, the Sperry method is not sensitive enough to detect hair-line cracks themselves so that rails so affected cannot be sorted out and scrapped at the mill; the rails must be in service long enough for the original hair-line crack to develop before its presence can be magnetically detected. The Sandberg controlled cooling process has consi-

derably reduced the risk of hair-line crack formation in rail-heads through controlling and retarding the rate of cooling of the product in a temperature range of 200°-400°C.

Published literature on this phenomena does not record occurrence of fracture in railway tyres. A number of other cases of transverse-fissure failure in tyres have been examined, each tyre steel conforming to standard specifications in respect of chemical and physical tests and free from metallurgical abnormalities. In a few cases the tyres that failed were fabricated from the same cast, which is in itself significant (*Iron & Coal Trade Rev.*, 1949, 158, 6).

### A New Austenitic Alloy

A NEW ALLOY FOR GAS TURBINE blades has been developed by a British firm. Known as G32, it is an austenitic alloy having a cobalt base and other main ingredients are nickel and chromium. It has also a small percentage of niobium.

G32 has been derived after 600 experimental melts. Further detailed tests are expected to show that it will be a worthy successor to G18B (hitherto predominant in the field of gas turbine discs). While, like G18B, primarily evolved for turbine engines, G32 has been shown to have other uses and is being produced for furnace parts such as cradles and structured members (*BIS*).

### Calcium Carbonate Extender Pigments

AN IMPORTANT CLASS OF EXTENDER pigments is the calcium carbonate class. A study of 5 calcium carbonate varieties, viz. dry ground calcite, wet ground calcite, "Witcarb regular" (medium-fine), fine-particle calcium carbonate and "Witcarb R" (ultra-fine) have been studied and the effect of particle size on the optical properties of the dried paint films and on the rheological properties of the fluid paint has been investigated (*Ind Eng. Chem.*, 1949, 41, 390).

The carbonates cover a particle size range from 0.5 to 3.9 microns, corresponding to specific surface areas from 0.32 to 0.55 sq. m. per gm. The oil absorption values increase with surface area, showing that surface area rather than dif-

ferences in the nature of the surface is the dominant factor. Formulations obtained with low acid value linseed oil also indicate that the particle size is primarily responsible for the variations observed in their properties.

Hiding power, lightness and gloss of a paint film increase regularly with decrease in particle size of the carbonate. Improvement in the above properties by the ultra-fine particle precipitated calcium carbonate appears to be due to improved dispersion of the binding pigment brought about by the calcium carbonate particles which may function as grinding or anti-flocculating agents.

Decreasing particle size of the calcium carbonate extender is accompanied by a regular increase in consistency and yield values of the paint.

### Potato Farming in the United States

WITHIN RECENT YEARS, THE average yield of potatoes per acre over the potato-growing territory in U.S.A. has more than doubled. In 1948 the average for the United States reached 204.5 bushels per acre. For these greatly increased yields, 4 developments are chiefly responsible: breeding of new varieties, better fertilizers, better sprays and the wide availability of certified seed stock.

Most important in the prevention of the spread of disease is the development of disease-resistant varieties of potato plants. Since 1932, under the national potato-breeding programme of the U.S. Department of Agriculture, at least 34 new varieties of various virtues have been distributed to the growers.

Of second importance in the control of disease at planting time is the programme of inspection and certification. A corps of government experts have the task of examining seed potatoes at various stages of their field growth and during the harvest, to the end that all seed potatoes they certify as acceptable will have, at most, only a tiny percentage of diseased plants.

The practice of crop rotation has been found to be a very effective additional control measure. If one or two crops of some other vegetation not susceptible to potato diseases is planted after the potato crop,

the disease organisms, or a large majority of them, die of starvation before they can affect the potatoes.

The fourth control measure, which is proving effective against the rhizoctonia, is the disinfection of the seed, either the whole tubers before they are cut, or the cut seed pieces. One recommended adjunct to disinfection is the suberization of seed pieces. This treatment causes the pieces to form over themselves a layer of cork cells which prevents their rotting in the moist ground. Since this method consists only of putting the seed pieces in a room with the proper humidity and heat for a short time, it is becoming a widespread and very rewarding practice.

The fifth control measure, now very common in areas where late blight and beetles harm huge numbers of the plants, is the spraying or dusting of the potatoes with various chemical mixtures. Among them is a DDT mixture which seems to have splendid effect on flea beetle, Colorado potato beetle, and leaf hopper.

In successfully raising seed potatoes which meet the stringent seed certification standards, farmers have taken advantage of a procedure which requires immensely more work at planting time but makes the rest of the growing and inspection of seed tubers much easier and safer. This is the "tuber unit" system of planting. All the seed pieces from single tubers are planted side by side, and a space is left at the end of each set of seed pieces before another tuber's pieces are planted. A corollary of this procedure is that the potato seed must be cut on the field at the time of planting, and after each set of seed pieces is planted, the knife used for cutting must be disinfected.

A good deal more care is necessary for this method of planting than when the farmer merely fills his seed hopper with pieces and starts off on his tractor. But once planted to tuber units, the crop becomes highly controllable.

Another necessary step in the programme of constant vigilance against potato diseases is "rouging", where diseased plants are removed by a close physical examination before they have chance to infect whole areas (*USIS*).

## Tropic Proofing

DURING THE WAR YEARS MUCH study and experiment was devoted to tropic proofing and to the packing of the equipment of every type used by Allied forces in Burma, Africa and other tropical theatres of war. The Directorate of Chemical Research and Development, Ministry of Supply, U.K., on the suggestion of the Department of Scientific & Industrial Research, have compiled in a booklet (H.M.S. Office, London; 1949, price 9d), the results of the extensive investigations of methods of protecting materials and equipment from deteriorative effects of storage and use in tropical climates. This publication is an up-to-date version of the war-time pamphlet published in 1945 and is now made available for general information of the civilian exporters to tropical countries.

The first 3 chapters in the pamphlet are devoted to methods of testing the efficiency of various treatments against fungal and insect attack. The remaining 8 chapters give information about the work done for the protection during storage and use in the tropics of various classes of materials and equipment, viz. plastics, rubber, leather, wood, textiles and cordage, paper and packing materials, paints and varnishes, lubricants and temporary metal preservatives and finally optical and electrical equipment. A list of Allied government publications is added as an appendix.

Brilliant sunshine, high temperature and humidity conditions and attack by molds and insects are the main causes of deterioration of the above type of materials and equipment in the tropics. Information relating to the choice of materials and the protective measures to be employed, including the use of appropriate fungicides and insecticides where necessary during use and storage, has been given under each head. The choice of any particular method of protection should generally be governed by a full consideration of all the factors likely to be involved and preferably after comparative trials. On the whole, the information contained in this pamphlet, although requiring to be supplemented by further details in many cases, is of immense practical interest

to manufacturers, traders and users of these materials and equipment in India.

The Defence Services in this country do already have a proper appraisal of the magnitude and importance of the problem of tropic proofing and have their own development organizations working on its different aspects, but it should be the task of the Indian Standards Institution to produce appropriate specifications for general adoption by the trade in India.

C. S. RAO

## Specifications for Radio Components

A SPECIFICATION DESCRIBING THE general conditions and procedure for durability testing of components for radio and other electronic equipment is published by the *Radio Industry Council*, U.K. The Specification No. RIC/11 is the work of the *R.I.C. Technical Specification Committee* in consultation with the *British Radio Equipment Manufacturers' Association*, the *Radio Communication & Electronic Engineering Association* and the *Radio Component Manufacturers' Federation* and is the first produced by the Committee. It covers approximately the same ground as the Inter-services Specification No. RC.S/11, but caters for the industry's requirements as distinct from those of the Services.

The object of the *Technical Specification Committee's* work is to produce a series of radio component specifications designed to ensure a high standard of reliability and performance for British components during use, transit and storage. Components are examined and their properties measured before and after they are subjected to the tests and their performances under test are laid down in the relevant component specifications. Components are classified under heads according to their ability to withstand extremes of temperature and humidity.

Supplementary design acceptance tests for vibration, salt atmospheric corrosion and mold growth are described.

Copies of the specification can be obtained (price 1s) from the *Radio Industry Council*, 59 Russel Square, London, W.C. 1.

## Television in Medical Training

FOR THE FIRST TIME IN EUROPE an audience of 200 medical

practitioners and students have been able to follow a surgical operation by television. This was on the occasion of the anniversary of Holland's oldest university at Leiden. Whilst an operation was being performed in the operating theatre of the hospital at Leiden, this was televised and projected on 2 screens of 1.30×1 m. set up in the lecture hall in another wing of the hospital.

Philips Eindhoven, who undertook the technical arrangement of this unique experiment in television after the most painstaking preparations, produced for the benefit of the audience a wonderful picture which demonstrated beyond a doubt the enormous possibilities of television as an aid to medical training.

## PATRA Research Laboratory

A NEW RESEARCH CENTRE WITH some 25,000 sq. ft. of floor area, to house the scientific and technological laboratories of the *British Printing, Packaging & Allied Trades Research Association* was recently opened at Leatherhead, in Surrey.

The PATRA laboratory stands on a 4.5 acre site and is designed and sited to allow further expansion. The building is approximately 200' long and 50' wide and of 3 floors. An outstanding feature is the technological laboratory which runs from the basement floor up to the top-storey floor level. This 20' of height and 2,000 sq. ft. of floor area will permit the installation of printing and packaging machinery. Letterpress and offset printing plants are installed here. Also at basement level are the laboratory for mechanical testing of packages, the process camera room and the electro-deposition and stereotyping department, and litho plate making section. To ensure constant working conditions, the atmosphere of the technological laboratory is controlled at 65°F. and 65 per cent relative humidity.

The package-testing laboratory is similarly controlled, but contains a variable humidity container conditioning the room with an operating range from 32°F. to 140°F. and 16 to 95 per cent relative humidity. The main laboratory here is equipped with rotating drums for testing the resistance of containers to rough handling with a drop-test apparatus.

Stores, boiler room and air-conditioning plant are housed in the basement.

At ground-floor level there is the entrance hall, flanked by a reception office and a committee room. Many of the scientific instruments used in PATRA's research work are designed and made in a workshop on this floor. The packaging research laboratory here contains a "make-up" table and equipment for making experimental packages and containers.

Paper and optics laboratories equipped for the study of the opacity, gloss and printing qualities of papers are also on the ground floor.

Study of the flow and deformation of inks and adhesives is carried out in the rheology laboratory (ground floor). The entomology, mycology and microscopy laboratories are also on this floor. Infection and damage to packages, books and papers by insects and molds, a major item on PATRA's research programme, is carried out here.

Scientific research on permeability in relation to packaging and packaging materials is the principal task of the packaging laboratory on the ground floor. Here, there is a variable high temperature room (for storage trials at elevated temperatures, designed to operate at controlled conditions between 65°F. and 140°F. and 10 per cent and 95 per cent R.H.) and a constant high temperature room (to operate at constant tropical conditions, 100°F. and 90 per cent R.H.).

Among the laboratories on the top floor of the new centre is that for surface chemistry where the desensitization of litho plates, contact angle measurement (for fundamental observations of the wettability of surfaces by oil and water), litho-plate-grain measurement, anti-corrosion processes for zinc and aluminium litho plates, and research into the influence of silica in desensitizing litho plates are among the tasks undertaken. On this floor is also the general enquiry laboratory for dealing with the workshop problems of individual members, and the laboratory dealing with absorption, oxidation and drying of printing inks.

Finally the building contains offices, a conference room, and the technical library — the key to which is a card index of over 100,000 items, covering books,

periodicals, pamphlets, patents, catalogues and other matter of informative interest to the printing and packaging industries.

### Multi-purpose Laboratory in the Himalayas

THE PROPOSAL FOR SETTING UP of a high altitude laboratory in the Himalayas was first mooted by the Government of India in 1947. The proposed laboratory will have separate departments dealing with the study of snow, glaciers and their contribution to Indian rivers, astronomical and astrophysical observations, cosmic rays, biological and geological research, and research on the flora and fauna of the Himalayas.

An exploratory party, sent out last year to select a suitable location, recommended a spot near Kauri pass, especially the 12,458' Ghorosan peak area which can be easily connected by a motor road with Joshimath, lower down on the route to the plains. 500' below Ghorosan lies a vast meadow known as *Aulivugyal* (pasture land) which would be suitable for the scientists' colony. The establishment of a hydro-electric station and arrangements for water supply would not present any insuperable difficulties at this spot.

A second exploratory party is being sent again by the Government of India this summer to gather more complete data before undertaking the project.

### Institute of Radio Physics

THE WEST BENGAL PREMIER, Dr. B. C. Roy, laid, on April 21, the foundation-stone of a Calcutta University institute which will, for the first time in India, provide for a post-graduate study and research in radio physics and electronics. Built with a capital grant of Rs. 5½ lakhs and an annual recurring grant of Rs. 49,000 from the Government of India, the institute will be located close to the institute of nuclear physics of the University College of Science. It is proposed to start in the institute new research schemes on microwaves, both fundamental and applied to television.

In recognition of the research work carried out by the University, the Council for Scientific & Industrial Research of the Commonwealth of Australia

have offered on permanent loan a complete set of ionospheric equipment to the University. A field-station for this apparatus would be built in the agricultural farm-land offered to the University by the Government of West Bengal at Haringhata, about 35 miles from Calcutta.

### Indian Roads Congress — 13th Annual Session, 1949

THIS SESSION OF THE CONGRESS was inaugurated by the Hon'ble Dr. Srikrishna Sinha, Premier of Bihar, on 4th February 1949 at Ranchi. 237 road engineers from all over India attended the session which was a pronounced success.

In his address, the retiring President of the Congress, Shri S. N. Chakravarti, stressed the important part that roads play in all development programmes, and referred to the many valuable contributions made by the Congress for the improvement of roads in all its aspects such as specifications, standards and design. He emphasized the importance of research on roads and welcomed the establishment of the Central Road Research Institute at Delhi. He advocated the establishment of a non-lapsing fund for road construction, avoiding waste of public money, and rendering possible for road engineers to complete their road development schemes according to programmes based on availability of resources. The President laid particular stress on the need for developing village roads for the benefit of the common man and the country as a whole.

9 committees and sub-committees dealing with specifications and standards, bridges, highway layout, research organization, soil research, test track, etc., held their meetings during the session and many technical papers were read and discussed.

Provincial chief engineers held a meeting to discuss programmes of road development in the different regions of the country, and measures for co-ordinating the work on national highways.

The exhibition organized by the Indian Roads Congress attracted much attention. The Roads Organization of the Government of India, Provincial Governments, the Indian Roads Congress Office, and firms dealing with road materials and machinery took part. Charts, maps

and statistical tables of road development programmes in India, soils and bituminous products, models of different types of timber bridges, etc., were exhibited. The Military Engineering Services had a separate section in which machinery and modern plant for road construction were on show. Considerable interest was evinced by the delegates in the mobile laboratory for bituminous road work exhibited by the *Shalimar Tar Products Ltd.*, Calcutta, and in the road rollers (steam and Diesel type) exhibited by the Government of Bihar.

Mr. S. M. Gross, Vice-President of the *International Division of the American Road Builders Association*, gave an interesting lecture on "Modern Drainage Practice in Highway Construction" and described some of the methods of bridging adopted on American roads.

Informative and instructive scientific films on road-building and road-making machinery added greatly to the interest of the road engineers who had gathered in full strength at Ranchi to discuss technical topics and exchange views on problems confronting them in their professional work. The Congress concluded with visits to the Barakar Bridge on the Grand Trunk Road and to the *Tata Iron & Steel Works* at Jamshedpur.

R. K. N. I.

### Committees on Geophysical Studies

AN IMPORTANT STEP FOR THE development of the study of geophysics in India has been taken by the appointment of 2 committees by the *Central Board of Geophysics*.

One committee, consisting of 5 members, will draw up a planned programme for geophysical studies in Indian universities and the scientific departments of the Government of India. Another committee of 7 will prepare plans of work regarding physical and biological oceanography covering as large an area as possible of the Indian seas.

The *Central Board of Geophysics*, which was constituted in March this year and held its first meeting recently, has decided to approach the Government of India for 5 overseas scholarships for specialized training in geophysics this year. Government will

be also approached for sanction for the establishment of magnetic and seismographic observatories at Dehra Dun in the immediate future. The establishment of a Central Geophysics Institute in India for advanced study and research in the science is also among the *Board's* plans.

### National Planning Committee

THE NATIONAL PLANNING COMMITTEE, which was appointed by the Indian National Congress 10 years ago, has completed its work. The report, which was adopted at the Committee's final meeting, has, as its appendices, the reports of its 29 sub-committees. The sub-committees' reports are published in 26 volumes, collectively known as the *National Planning Committee Series*.

The Committee defined its own objectives, laid out a bold code of wide instruction for the guidance of these sub-committees and prescribed clear-cut norms and stages for the completion of its comprehensive task.

The reports of the 29 original sub-committees and 2 *ad hoc* sub-committees deal with the following: (1) manufacturing industries; (2) chemical industries; (3) population; (4) trade; (5) power and fuel; (6) women's role in planned economy; (7) rural marketing and finance; (8) labour; (9) river training and irrigation; (10) animal husbandry and dairying, fisheries and horticulture; (11) national housing; (12) education (general and technical); (13) communications; (14) engineering industries and industries connected with scientific instruments; (15) mining and metallurgy; (16) soil conservation and afforestation; (17) insurance; (18) land policy and agricultural, labour and insurance; (19) rural and cottage industries; (20) industrial finance; (21) crop planting and production; (22) currency and banking; (23) public finance; (24) transport services; (25) national health; and (26) national planning, its principles and administration.

### Central Silk Board

THE GOVERNMENT OF INDIA HAVE set up a Central Silk Board to provide for the development under central control of the raw silk industry in the country. The Board will advise the Central Government on all matters relating to the raw silk industry

including the import and export of raw silk. Among its other functions, the Board will devise means for improved methods of mulberry cultivation, rearing, developing and distributing healthy silk-worm seed, reeling and the supply of technical advice to filature and *charkha* reelers and help to improve the marketing of raw silk.

The Board will consist of 27 members with the Minister for Industry and Supply as Chairman. By the Act under which the Board is constituted, it is empowered to levy a cess on the silk industry.

### Agricultural Census

IN PURSUANCE OF A RECOMMENDATION made by the *FAO* for organizing an agricultural census in 1950 in member-countries, the Government of India have appointed a technical committee consisting of Mr. W. R. Natu, Economic and Statistical Adviser, Ministry of Agriculture, as Chairman and the following as members: Prof. P. C. Mahalanobis, Statistical Adviser, Cabinet Secretariat, New Delhi; Dr. P. V. Sukhatme, Statistical Adviser, Indian Council of Agricultural Research; Dr. V. G. Panse, Deputy Director (Research), Institute of Plant Industry, Indore; Dr. B. Natrajan, Economic Adviser to the Government of Madras; Mr. G. M. Sankpal, Director, Bureau of Economics and Statistics, Government of Bombay; Mr. K. Kishen, Statistician to the Department of Agriculture, United Provinces; Mr. N. C. Chakravarty, Department of Agriculture, Forests and Fisheries, Government of West Bengal; Mr. H. C. Kothari, Statistical Officer, Government of Jaipur; Mr. K. S. Sursinghi, Director of Agriculture, Government of Saurashtra; Mr. B. A. Bombawale, Secretary to the C.P. Government Land Records Department; and Mr. J. S. Sharma, Research Officer, Directorate of Economics and Statistics, Ministry of Agriculture (Secretary).

The terms of reference of the committee are: (a) to examine the forms of returns at present used by the different provinces and States for routine collection of data and evolve standard forms of returns; (b) to consider whether any special modifications are necessary in the scope of enquiry in respect of areas where the system of land survey



record of rights is not introduced ; (c) to explore the directions in which the data collected through the census of agriculture can be usefully integrated with the data collected through the census of population.

### Village Oilseeds Industry Board

THE INDIAN OILSEEDS COMMITTEE, at its 4th annual general meeting, recommended the setting up of a village oilseeds industry board and a training institute at Nagpur for the development of the village oil industry. The board named *The Ghani Advisory Board of the Indian Oilseeds Committee* will study the progress of the schemes sanctioned for *ghani* development and organization of co-operative societies. It will also supervise the activities of the training institute.

To encourage crushing of seeds in village *ghanis* the Committee requested the Provincial Governments to adopt a uniform policy with regard to exemption limits from sales tax, and suggested that the limit should be Rs. 15,000 in all the provinces.

### The Metal Market Review— 1949 Annual Number

TO MARK THE COMPLETION OF the first year of its publication the *Metal Market Review* has brought out a well got-up and highly informative special annual number ( Vol. 1, No. 25, 1st Feb. 1949). The issue contains a series of articles written by well-known authorities in various branches of metallurgical industries and metal trade.

The number is divided into 4 parts. The first part deals with topics of general interest pertaining to metal trade and non-ferrous metallurgical industry. The second part is mainly concerned with the ferrous metallurgy. The third part contains valuable statistics. The last part

is a useful "who is who" in metal trade and industry.

### Grant for Andhra University

THE GOVERNMENT OF INDIA have sanctioned a grant of Rs. 770,000 to the Andhra University non-recurring, and Rs. 88,000, recurring, for the expansion and development of the Technology Department including the organization of a new branch in pharmaceuticals. The buildings are estimated to cost Rs. 380,000 and the equipment Rs. 390,000.

### Grant for College of Technology, Madras

THE GOVERNMENT OF INDIA sanctioned for the Alagappa Chettiar College of Technology, Madras, a sum of Rs. 2,92,000 for the college buildings, and Rs. 1,13,000 for equipment. They have also sanctioned for the 2 departments of Chemical Engineering and Textile Technology an ultimate recurring grant of Rs. 40,000 and 32,000 respectively. The grants are made on the understanding that the number of admissions will be increased and the admissions will be made from candidates applying from different provinces.

### Announcements

*Grant for Preparation of History of Chemistry in Ancient and Mediaeval India*—The Government of West Bengal have contributed a sum of Rs. 5,000 towards the preparation of the "History of Chemistry in Ancient and Mediaeval India" (incorporating Sir P. C. Ray's "History of Hindu Chemistry") undertaken by the *Indian Chemical Society*.

*The Indian Standards Institution* has issued the following 5 Draft Indian Standards for comment by those interested in them: Two standards on Mica; Coniferous Timber; Procedures for Testing Cotton Textiles and

Cordages for Resistance to Attack by Micro-organisms; and Draft Indian Standard Style Manual. These can be obtained by application to the Secretary (Publications), Indian Standards Institution, Block No. 11, Old Secretariat, Delhi.

Comments on these Standards should reach the Director, Indian Standards Institution, Block No. 11, Old Secretariat, Delhi, by June 30, 1949.

## REPORTS FROM STATES & PROVINCES

( continued )

cultivators as a winter crop in central districts and for conducting trials of Sea Island varieties on the west coast. The target is to evolve a strain of staple length 11/16" and 1 1/4" over the 4.5 lakh acre winter crop area, yielding 4 lakh bales and 6 lakh acres in the west coast districts under Sea Island cotton yielding 1 lakh bales of 1 1/4" to 1 1/2" staple length.

### Cycle Factory

THE GOVERNMENT OF INDIA HAVE provisionally agreed to the establishment at Madras of a cycle factory by *Messrs B.S.A.* bicycle manufacturers with a capacity of 1 lakh cycles per year.

Permission has also been granted for the setting up by a British firm of a plant for the manufacture of earth-moving machinery used in building roads and dams.

## UNITED PROVINCES

### Soil Laboratory

THE AGRICULTURE MINISTER OF the province opened the Regional Soil Laboratory at Aligarh on April 21. The laboratory, the first of its kind in India, will undertake soil survey for the benefit of agriculturists.

# Reports from States & Provinces

## MYSORE

### Manufacture of Telephones

THE TELEPHONE INDUSTRIES Ltd., Bangalore, has gone into production and is assembling and finishing telephone instruments at the rate of 300 per week.

The factory is now housed in 2 huge hangars on the site of the proposed building. An extensive plot of 365 acres have been cleared on the Bangalore-Madura Trunk Road for putting up the factory buildings and residential quarters. There will be 8 huge workshop sheds with administrative blocks. Water and light have been laid. To facilitate quick transmission a railway siding is provided. The buildings are to be completed by 1951. The factory when in full swing is expected to provide employment for 10,000 persons.

### New Engineering College

DAVANGERE, AN IMPORTANT commercial centre and third largest city in Mysore State, is to have an engineering college from the coming academic year. The Government have also sanctioned the establishment of a polytechnic in this city.

## BIHAR

### Power Alcohol Plant

THE REVENUE MINISTER OF Bihar inaugurated the biggest power alcohol plant in India at Mirganj in Saran district on April 13. The plant has a daily capacity of 3,000 gal.

## BOMBAY

### Penicillin Factory

THE RS. 3 CRORE PROJECT FOR the manufacture of penicillin, sulpha drugs and anti-malarials was approved at a recent conference held in Bombay and presided over by Dr. Syama Prasad Mookerjee, Minister for Industry and Supply, Government of India. The Conference approved of the suggestion to locate the penicillin factory at Dehu Road, 16 miles from Poona.

## NEW DELHI

### Machine Tools Factory

NEGOTIATIONS FOR AN AGREEMENT with the *Oerlikon Machine Tools Buchle & Co.*, Zurich-Oerlikon, Switzerland, for the establishment in India of a factory to manufacture machine tools have been successfully concluded. The capital cost of the factory is estimated at Rs. 12 crores and the value of the production at Rs. 7 to 8 crores a year. The factory is expected to be completed within a period of 4 years.

The agreement provides *inter alia* that the Swiss firm will render all technical assistance in the erection of the factory, training of Indian personnel and direction and supervision of the production of the factory for 20 years from the date the production starts. They will also have a small financial interest in the project.

The Swiss firm will train Indian workmen, engineers and other technical personnel required for the factory in Switzerland. The Government of India will meet only the travelling and living expenses of these trainees and no fees for training will be charged. The firm will also set up a training school in India for the purpose of training Indian workmen, engineers and other technical men required for the factory and provide the necessary expert personnel and equipment to train such men. They will endeavour to train Indian personnel in such a way that within a period of 10 years from the date of the starting of the factory there would be sufficient number available to hold not less than 85 per cent of the technical posts in the factory.

## MADRAS

### Millet Research

THE OPENING OF FOUR MILLET research stations has been sanctioned by the Government as a temporary measure in the scheme for making the province self-

sufficient regarding millets. Narasapatam in Vizagapatam district, Ongole in Guntur district, Tirupattur in North Arcot district, and Ariyalur in Tiruchirapalli district will be the places at which the new research stations will be established.

The work of these stations will be to produce high-yielding millets suitable to different parts of the province within a period of 5 years. The cost of the scheme is estimated at Rs. 2 lakhs.

### Paper Research

THE GOVERNMENT HAVE SANCTIONED a scheme for establishing a paper research station in North Malabar for the purpose of giving scientific assistance to paper industry in South India. The scheme will run for a period of 4 years in the first instance. The station is to be located near Payyanoor.

### Limestone Deposits

A REPORT ON THE MINERAL resources of Tiruchendur and Nanguneri taluks of Tirunelveli district shows large deposits of good limestone estimated at 9,000,000 tons in Sattankulam area. This area is ideally suited for the location of cement industry also.

### Ergot Production in the Nilgiris

THE SCHEME FOR THE EXTENSIVE production of ergot on the Nilgiri Hills has been extended by the Government for a further period of 3 years from the current year. The subventions to the cultivators of rye has been increased to Rs. 10-12-0 per lb. of dry ergot, in addition to the free supply of culture for spraying and the necessary equipment. Since the inception of the scheme in 1944 the Agriculture Department has produced 5,745 lb. of ergot.

There has been considerable improvement in the quality of ergot produced. In 1944-45 when the scheme started, the alkaloid content of ergot was only a little over 0.20 per cent and at present it has gone up to nearly 0.45 per cent.

### Growing of

### Long-staple Cotton

THE GOVERNMENT HAVE SANCTIONED a 5-year scheme for the evolution of a suitable strain of long-staple American cotton for

(continued on page 252)



# INDIAN PATENTS

The following is a list of a few of the Patent Applications accepted in the *Gazette of India*, Part II, Section I, for April 1949.

## Plastics & Plasticisers

38615. I.C.I. LTD.: Improved anion exchange resins: *Subjecting a chlorinated paraffin wax and an alkylene polyamine to an elevated temperature.*

## Inorganic Chemicals

37423. DELHI CLOTH & GENERAL MILLS CO. LTD., SHARMA, JAIN & CHIPALKATTI: Process for the conversion of titanium into soluble state from materials containing titanium such as bauxite sludge: *Bauxite sludge in dried form is ground and mixed with sulphuric acid and heated.*
40185. ANGLO-IRANIAN OIL CO. LTD.: Catalytic desulphurization of a petroleum fraction: *Passing the distillate vapour over a cobalt-molybdate-alumina catalyst at 700°-800°F. at a pressure of 250 lb./sq. in.*
40505. N. V. ZUID-NEDERLANDSCHE SPIRITUS-FABRIEK: Method of working up waste liquor of fermentation industries in which molasses are used: *Partly evaporated liquor is burnt to reduce the potassium sulphate to sulphide and finally treating the sulphide with carbon dioxide to obtain potassium carbonate.*

## Organic Chemicals

38339. SHARP & DOM INCORP.: Preparation of benzoic acid esters of secondary-alkyl (secondary) amino-propanols and butanols and the products of said methods: *Reacting benzoic acid anhydride or halide with secondary alkyl amino propanol or butanol.*
38502. I.C.I. New leuco sulphuric esters of vat dyestuffs: *Forming the leuco sulphuric ester in the presence of organic amide in which hydrogen atoms attached to amide nitrogen have been replaced by hydrocarbon radicals.*
38554. I.C.I. LTD.: New dyestuffs of the anthraquinone series: *Sulphonating the product obtained by reacting 2: 3-dichloro-1: 4-diamino-anthraquinone with an alkali metal-phenoxide varying a phenyl group in para position.*
38555. I.C.I. LTD.: Vat dyestuff: *Sulphonating in the presence of organic amide in which hydrogen atoms attached to amide nitrogen atom have been replaced by hydrocarbon radicals.*
38556. I.C.I. LTD.: New leuco sulphuric esters of vat dyestuffs: *Sulphonating in the presence of an organic amide in which hydrogen atoms attached to amide nitrogen atom have been replaced by hydrocarbon radicals.*
38658. I.C.I. LTD.: Vat dyestuffs: *Sulphonating naphthalthioindigoid dyestuffs which contain chloro and/or nitro groups in the naphthalene nuclei.*
38690. LES USINES DE MELLE: Process for the production of acetone and butyl alcohol by fermentation: *A non-fermentable acid is supplied to fermenting mash by an amount*

*not higher than equivalent of the amount of neutralizing agent added.*

40175. N.V. DE BATAFSCHE PETROLEUM MAAT-SHAPPIJ: Process for producing alcohols: *Reacting ethylene with water in the presence of a catalyst latter consisting of activated bentonite clay impregnated with hydrofluoric acid.*

## Food & Kindred Products

40544. LEVER BROTHERS & UNILEVER LTD.: Manufacture of margarine: *Successively warming, cooling and subjecting margarine to mechanical working.*
28820. BROWN: Grain flour: *Temperature of cereal maintained below the brittle point of flour cells from the time it is tempered until final milling operation.*

## Instruments

37154. DAKSHINAMURTHY: Humidity measurement devices: *Circuit consisting of wet and dry thermocouples and one or more galvanometers with switch means to connect the thermocouples selectively with the galvanometer.*

## Leather & Leather Products

40231. NEUMANN: A process for preserving hides and skins: *After usual liming, bating and scudding the pelts are treated with preservative comprising common salt and sodium metaphosphate.*

## Medical Research & Practice, Drugs & Pharmaceuticals

39121. MARCELLE FLEISCHMANN FOUNDATION INC.: Penicillin preparations: *Comprising calcium penicillin in beeswax and peanut oil.*
40233. MERCK & CO. INC.: Processes for manufacturing therapeutically valuable salts of penicillin G: *Reacting in solution an amine salt of penicillin G and a salt of alkali metal or alkaline-earth metal salts.*
40469. MERCK & CO. INC.: Procaine salt of penicillin: *Reacting penicillin or a salt of penicillin with procaine or a salt of procaine.*

## Metals & Metal Products

38508. METALS-GAS CO. LTD. & R. HUNTER: Processes for coating a metal or alloy: *Passing a continuous stream of a gaseous halide of the coating metal over the metal to be coated.*

## Miscellaneous

40957. JANSEN: Ceiling boards: *Having coconut fibre dust as base.*
38683. I.C.I. LTD.: Seed dressing: *Comprises gamma-benzene hexachloride and an organic mercurial seed disinfectant.*
38845. I.C.I. LTD.: Seed dressing: *Consisting of gelling clay mixed with water soluble dispersing agent and an organic mercury seed disinfectant.*

## Natural Resources & Human Welfare

**R**ECENT discussions on the utilization of natural resources among scientists and economists reveal a refreshing shift of emphasis from "exploitation" to "conservation". Exploitation was the dominant feature of resources utilization during the war years when "working plans" were discarded and "slaughter tapping" was resorted to. The very ruthlessness of the exploitation led the thinking section of the people — among them scientists, technologists and economists — to pause and consider the consequences of current practices on the life and productivity of resources which foster and sustain life. It was indeed appropriate that the Secretary General of the *United Nations Organization*, following the discussions of the Economic and Social Council (March 28, 1947), decided to call, in August 1949, a Scientific Conference on the Conservation and Utilization of Resources with a view to enable all those who have given thought to the subject to exchange information on techniques in the field, their economic costs and benefits, and their inter-relations. The problem has rightly assumed international importance, for it transcends national and group interests and affects the welfare of mankind as a whole. Concern has been expressed in various quarters that the lack of balance between growing populations and waning resources is attaining increasingly alarming proportions, and that such lack of balance would lead to widespread starvation and famine. Since the decision to hold the international conference was taken, an Inter-American Conference on Conservation of Renewable Natural Resources was convened at Denver, Colorado (September 7-20, 1948), at which representatives of 21 nations examined the conservation problem at the technical level. The papers discussed at the working sessions of the Conference emphasized the magnitude of the problem

and the need for continuing co-operation among nations to ensure effective action towards the solution of a problem of such vital concern to the human race.

The Declaration of the Inter-American Conference indicates the importance of the problem. "The crucial problem of our generation", it states, "is to safeguard, maintain, develop, increase, and wisely use for the common benefit of mankind the natural resources of the earth... The catastrophe that threatens civilization results from man's failure to live in harmony with the principles that govern his environment."

For the effective handling of the problem, it is necessary to understand the "principles that govern man's environment". History furnishes many examples of civilizations which flourished for a time and then decayed largely as a result of a lack of harmony between the people and the environment. A historical study of the systems of resources utilization at various chronological epochs of man's evolution should prove instructive and helpful to the proper appreciation of the factors that determine man's prosperity and decay. It is important to recognize that man lives in "an environment of many facets, related to each other, not as single pieces but as a mosaic, the pattern of which is not easily discerned at first glance. It must be seen in different lights before we appreciate its full design and real worth." \* For his very existence man must interfere with nature and in this act he disturbs one or more pieces of the mosaic whose consequences, if not foreseen and promptly remedied, may lead to disastrous consequences. Resources utilization, therefore, must be based on a clear understanding of the ecological factors constituting the environment.

\* *Nature, Ecology in Service of Man (Rev.)*, 1945, 156, 675.

### Unified Resources Development

In the past, when populations were sparsely distributed, nomadic existence was the rule, and resources were relatively abundant, there was little urgency for resorting to conservation practices. With the growth of population and the coming into existence of settled life, the pressure on resources necessarily increased. The resources problem was aggravated by a perceptible diminution in the productivity of the land and the sea. Industrialization and urbanization gave rise to vested interests and pressure groups with pronounced tendencies to exploit resources oblivious to consequences. It became clear that resources utilization must receive prior attention if the productivity of what was left is to be sustained and the needs of future generations are to be provided for. Specific short-term corrective devices were inadequate and ineffective, and a scientific approach to the management problem, which took into account the ecological factors, was called for. Long-term programmes of a constructive and restorative character provided an effective safeguard against permanent damage to productivity. Water, soil, woods, forage, and wild life comprise a "biotic whole" in relative equilibrium, the loss of which may result in the ruin of any or all of its constituents.\* Unified resources development, which takes into cognizance the complex nature of the biotic balance, offered a plausible solution. How this can be achieved on a regional basis was amply demonstrated under the leadership of the late United States' President, Theodore Roosevelt, in the Tennessee Valley. This successful experiment stands out as an object lesson to the nations of the world, and as an example of the tactics and strategy of resources conservation and management.

### Planning on a National Basis

In considering national economic programmes, it is necessary to examine current utilization practices in terms of their effect on future resources capacities. The questions that demand consideration are: Are we using our national wealth faster than it is being renewed? What are the principles of management which ensure sustained productivity of renewable resources? Scientists in their approach to the conservation problem

are inspired by the faith that the world's resources could be developed to take care of the economic needs of the people to a greater extent than in the past. Every nation is concerned with maximizing production, and programmes for so doing have received the highest priority. How this can be done even in countries with limited *per capita* resources is a problem which should receive the earnest consideration of scientists and administrators alike. In post-war Japan, for instance, some of the most intricate problems of occupation by Allied Forces involved the technology of resources utilization. The National Resources Section, which was established in Tokyo immediately after the cessation of the war as a special Staff Section of the G.H.Q. of the Supreme Commander for the Allied Forces, has tackled the problem of Nippon's shattered economy with conspicuous ability. At the time the National Resources Section was set up, the position appeared somewhat gloomy. Farm lands had been intensely cultivated for centuries and their fertility had greatly diminished. Fisheries and forests had been over-exploited. The wealth of the land and the sea was concentrated in the hands of a relatively few individuals. Sweeping reforms in ownership were introduced. Reconnaissance surveys were made and a general inventory of resources was taken. Statistics were collected. Japan's science and technology were built on European and American foundations but the liaison between them was poor. Research and development had to be reorganized to produce practical results. Economic planning had to be considered on a national scale, and the National Resources Section, with vision and knowledge and armed with reliable factual data, evolved working plans for the utilization of both renewable and non-renewable resources. "What is being done in Japan to build a good society, economically, socially, and politically may, if it is successful, serve to justify the efforts and sacrifices of millions of people in fighting this part of World War II."\*

### Conservation must be made Effective

The success of utilization planning in the Tennessee Valley and in Japan are significant, and serve as pointers to what is possible to achieve in other regions of the world. No country can afford to be complacent about

\* Cf. H. P. SALAZAR: *Woods and Pastures*, Inter-American Conference on Conservation, p. 61.

\* *Science*, 1948, 108, 367.

its natural resources, and no country has yet formulated or adopted a national policy of resources management for maintaining their productive potential to perpetuity. What has been done only shows what remains yet to be done. It is fortunate that in India poverty and ignorance in the past had stood in the way of exploiting, or even mapping, of *all* her resources, and there is every hope that a planned utilization of the resources would lead to increased prosperity and a higher living standard for her people. The initiative taken by the Government for the multi-purpose development of water resources gives hope that the problem of constructive management is beginning to receive attention. Scientists have the responsibility of not only taking a full inventory of renewable and exhaustible resources, but also of evolving sound standards of resources management, and of influencing national policies. They have the even greater responsibility of educating the public and making it conservation-conscious, for no plan, however sound, has any chance of becoming effective unless the people are receptive and responsive to

its principles and techniques. Conservation should become a part of the social philosophy of the people.

It is often said that the most serious catastrophe facing humanity is slow starvation. This catastrophe can be averted if resources utilization is managed on a sustained yield basis. Science provides the basis for such plans and programmes. What is needed is the determination to apply the results and methods of science for promoting human welfare. By enabling the leading scientists and science administrators from all parts of the world to meet in conference\* and discuss conservation programmes and policies, the *United Nations Organization* has rendered a momentous service to the cause of peace and prosperity of mankind.

\* A scientific delegation led by Dr. Shanti Swarup Bhatnagar and composed of the following members has been sponsored by the Government of India to take part in the forthcoming conference to be held at Lake Success: Dr. S. S. Bhatnagar, Dr. J. C. Ghosh, Dr. J. N. Mukherjee, Dr. P. C. Mahalanobis, Dr. M. S. Krishnan, Dr. S. L. Hora and Mr. A. N. Khosla.

## Characteristics of the Ionosphere over Calcutta (April 1949)

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(Miss) T. K. PAL & A. P. MITRA

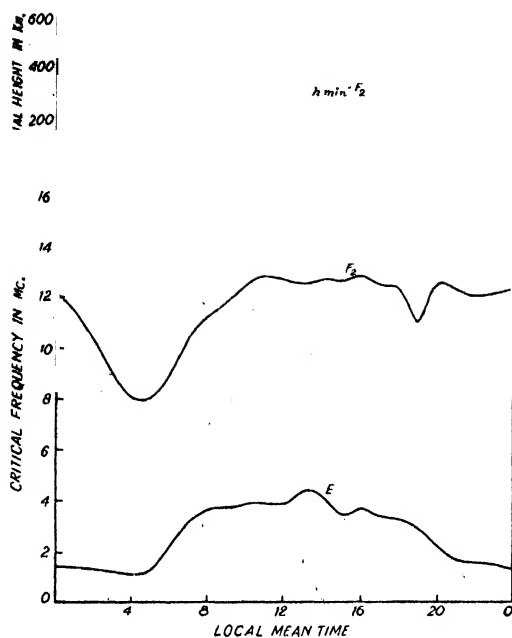
*Wireless Laboratory, University College of Science, Calcutta*

THE following are the ionospheric data observed at Calcutta for the month of April 1949. The observations were made at each hour of the day for 5 days a week.

Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form. The figure represents the mean of both normal and abnormal values. The special feature

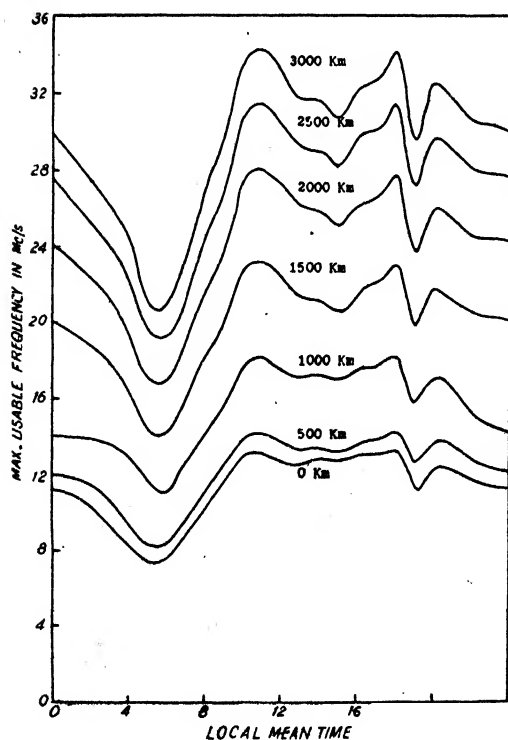
of the  $F_2$  region during the month is that the ionization maintains a high value from about 10 a.m. till midnight.

Fig. 2 gives the predictions of maximum usable frequencies which can be used for different distances of transmission during July 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when the E region ionization was found to be abnormal and the corresponding penetration frequencies and heights.



( 5 HOURS 54 MINUTES AHEAD OF G.M.T. )

FIG. 1 — APRIL 1949.



AT POINT OF REFLECTION.

FIG. 2 — PREDICTED M.U.F. VIA  $F_2$  LAYER, JULY 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ * Mc.	$hE_s$ Km.
April 1949	*11	21.00	5.25	105
		22.00	4.25	105
		23.00	6.00	105
	12	08.00	5.75	120
		09.00	6.60	135
		23.00	2.65	90
	13	00.00	2.00	90
		06.00	3.50	105
		07.00	3.85	105
		08.00	4.65	120
		10.00	5.20	120
	*14	13.00	5.20	150
		15.00	5.00	150
		16.00	5.70	150
		17.00	4.00	135
		20.00	6.75	135
		21.00	4.00	120
		22.00	4.00	120
	*15	07.00	6.50	150
		08.00	9.60	150
		09.00	6.65	150
		11.00	6.00	150
		12.00	6.65	150
		13.00	6.25	150
		14.00	5.80	150
	*16	09.00	7.15	150
		10.00	7.00	150
		11.00	5.80	150
	18	21.00	2.50	105
		22.00	4.00	105
		23.00	3.25	105
	19	00.00	3.00	90
		01.00	3.00	90
		02.00	2.80	90
		07.00	4.25	105
		08.00	5.75	120
		09.00	5.25	120
		20.00	5.00	120
		21.00	4.25	120
		22.00	3.50	120
		23.00	3.30	105
	20	00.00	3.00	90
		06.00	6.00	120
		07.00	3.50	120
		09.00	5.00	135
		11.00	5.00	135
	*21	09.00	4.20	135
		11.00	5.20	150
		12.00	5.50	150
		19.00	6.45	135
	25	18.00	3.40	105
		19.00	3.50	105
		20.00	3.00	90
		22.00	3.50	90
		23.00	2.75	90
	*26	07.00	3.50	120
		08.00	4.25	120
		09.00	5.25	135
		10.00	6.00	150
		11.00	6.25	150
	*27	07.00	5.25	135
		09.00	5.15	135
		10.00	5.25	135
	28	09.00	11.00	150
		10.00	8.70	150
		20.00	3.25	105
		21.00	2.70	105
	*30	08.00	4.75	120
		09.00	9.75	135

\* Local thunderstorm.

# A Cosmic Ray Radio Sonde

R. P. THATTE

*Tata Institute of Fundamental Research, Bombay*

MILLIKAN, Neher and Pickering<sup>1</sup>, in 1939-40, investigated total cosmic ray intensity in India by using radio sonde technique, and G-M counters singly or in coincidence telescopes. They made a latitude survey extending from Madras, 3°N., to Peshawar, 25°N. geomagnetic latitude. The geometry of their coincidence telescopes was such that their data can be compared only with great difficulty with any other high altitude data using telescopes of different geometry. At the *Tata Institute of Fundamental Research*, a programme was, therefore, undertaken to carry out an accurate high altitude survey with telescopes of standardized geometry, both with and without lead absorber, at several different latitudes varying from the magnetic equator northwards. In August and September 1948, seven high altitude soundings were made at New Delhi, 19°N. magnetic latitude, as a preliminary to standardizing the technique. The results obtained will be published separately by Messrs A. S. Rao and G. S. Gokhale. The purpose of this paper is to describe the apparatus used in these flights.

## The Equipment

Generally speaking, the radio sonde technique, when applied to high altitude cosmic ray investigations by unmanned balloons, consists essentially in transmitting the cosmic ray data, along with the pressure-temperature data, over an ultra-high frequency carrier from the balloon-borne equipment to the ground station where it is received and recorded. The equipment used, therefore, will be described in two parts, namely the equipment that goes up and the equipment on the ground.

## The Balloon-borne Equipment

This consists of a telescope of chosen geometry using G-M counters in coincidence; the coincidence circuit; the transmitting circuit; the meteorograph, which sends the pressure-temperature data, from which the height of the apparatus above ground at

any instant during the flight can be computed; the necessary H.T. and L.T. supplies for the electronic circuits, and the H.T. supply for the G-M counters.

*The Counter Telescope* — It was decided to use four counters in quadruple coincidence instead of the triple coincidence generally used, to reduce the relative count due to side showers. Fig. 1 is the coincidence circuit of the conventional Rossi type. The four, 1-N-5-GT-G, 1.5 volt filament, sharp cut-off R.F. pentodes form the four Rossi coincidence tubes. The centre wire of the G-M counter is directly connected to the grid of the coincidence tube, its 1 megohm grid leak forming the load resistance of the self-quenching counter. The coincidence anode load resistance is 1 megohm. All the screens of the coincidence tubes are connected together and connected to the 90 volt H.T. anode supply through a 220K ohm resistor. Although this direct connection to the coincidence valve grid and the unconventionally high anode load resistance are rather unorthodox, they were found to give large discrimination ratios of the true to false coincidences. For example, using a pulse generator with three tubes receiving a common pulse and the fourth having no input, a 140 volt pulse was required on the three grids to give a coincidence pulse of the same height as obtained with only a 4 volt pulse in a true quadruple coincidence. Similarly, it was found that the ratio of pulse input for only two tubes, the others floating, to the true quadruple coincidence pulse input was still higher than above.

Fig. 2 is a photograph, showing the coincidence circuit mounted on the counter telescope frame.

*The Resolving Time of the Coincidence Circuit* — The resolving time of a coincidence circuit has to be low to reduce the accidental error. Using the data of Neher and Pickering<sup>2</sup> obtained at Agra, 17°N., the total intensity of a single counter will be approximately 50 times the ground intensity at the maximum at high altitudes. Also, the

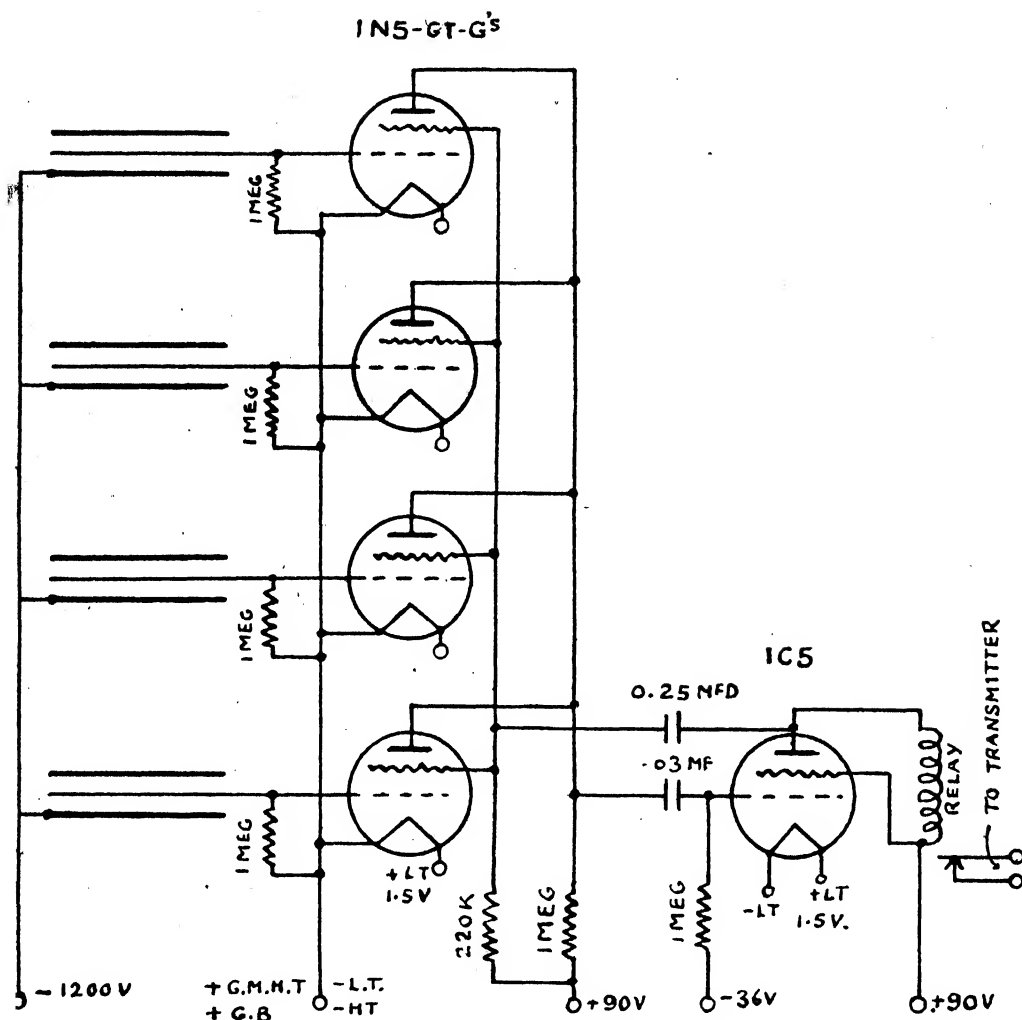


FIG. 1 — THE COINCIDENCE CIRCUIT.

intensity of a quadruple coincidence telescope will be approximately 20 times the ground intensity at the maximum. For the counters used by us, this would give the rate for a single counter of 150 counts per second, and 0.25 per second for the quadruples at the maximum.

The resolving time for the above coincidence circuit was measured by the usual method of measuring the accidental rate and using the formula

$$A = 4N_1N_2N_3N_4\tau^3 \text{ — for quadruple}$$

coincidence, where  $A$  is the accidental rate and  $N_1$  to  $N_4$  are the counting rates of the four individual counters placed in such a way that no single particle can pass through all

of them. The value of  $\tau$ , the resolving time, was  $7.3 \times 10^{-5}$  sec.

Thus, at the maximum counting rate of a single counter, the accidental rate, calculated from the same formula for the above resolving time, would be  $7.87 \times 10^{-4}$  counts/sec., which would give an error of only 0.31 per cent at the maximum expected quadruple rate of 0.25/sec. The error would be much lower for lower counting rates and, therefore, negligible.

The output circuit of the coincidence unit is similar to that used by Neher and Pickering<sup>3</sup> with screen feed-back. With the time constants of the feed-back circuit as shown, the width of the output pulse is 0.05 second. The output stage has in its

anode circuit a high-speed sensitive relay. This valve is normally kept at plate current cut-off by the 36 volt grid bias; the relay is in an unenergized state, and the normally closed pair of its contacts is inserted in the plate H.T. circuit of the coincidence transmitter. The transmitter, therefore, gives a continuous carrier when there is no coincidence pulse. When the shaped pulse opens the relay contacts for 0.05 sec., the transmitter is switched off for this duration. Since the counting rate of our telescope was low, this method of having the carrier continuously on, except for the duration of the coincidence pulse, was found to be a great help in keeping the receiver sharply tuned to the carrier.

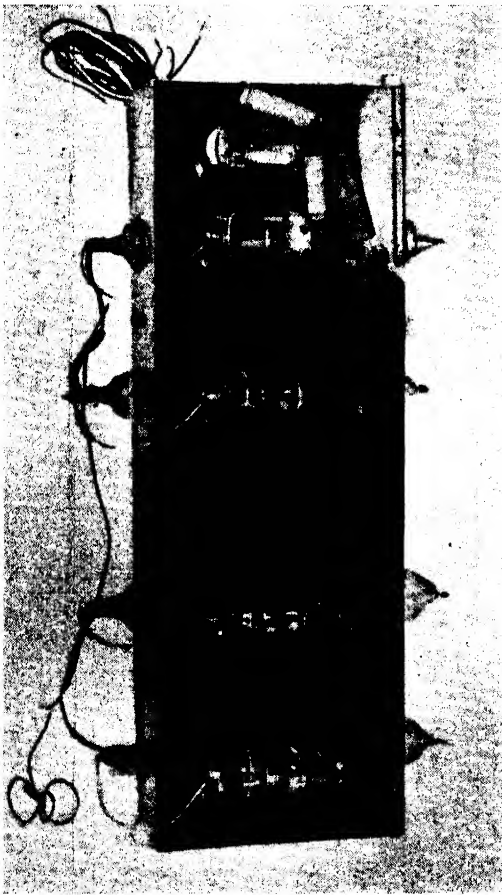


FIG. 2 — THE COINCIDENCE TELESCOPE — THE COINCIDENCE CIRCUIT IS WIRED ON A BAKELITE PANEL FORMING ONE SIDE OF THE TELESCOPE FRAME. NOTE THE SHORT CONNECTIONS FROM THE COUNTER ANODES TO THE VALVE GRIDS. THE VALVES ARE DEBASED AND WIRED ON THE PANEL.

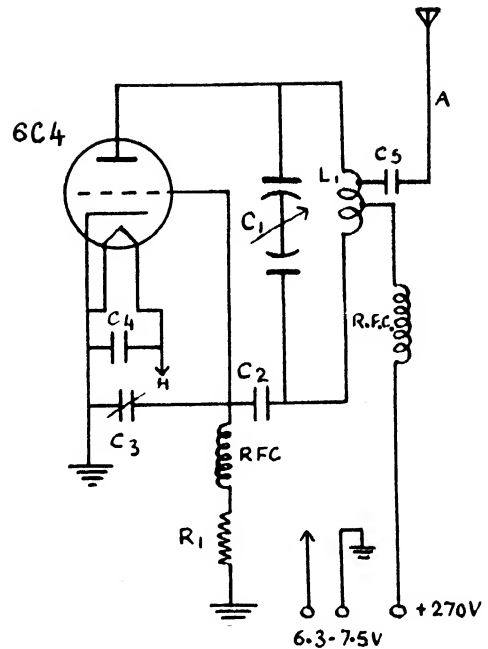


FIG. 3 — THE U.H.F. TRANSMITTER CIRCUIT — THE VALUES OF THE DIFFERENT COMPONENTS USED ARE AS UNDER:  $C_1$ —Split stator tuning condenser.  $C_2$ —50 mmfd. midget mica.  $C_3$ —3-30 mmfd. ceramic trimmer.  $C_4$ —500 mmfd. midget mica.  $C_5$ —50 mmfd. silver mica.  $R_1$ —10,000 ohms grid leak.  $L_1$ —2 turns, 10 swg 0.7"—151 mc.; 2 turns, 10 swg 1.0"—141 mc. RFC—1 inch winding, 24 swg enamelled on  $\frac{1}{4}$ " diameter polystyrene rod. A—half wavelength aerial.

*Errors Due to the Resolving Time of the Carrier Pulse*—For the duration of the carrier pulse of 0.05 sec., the relative error introduced at the maximum counting rate of 0.25/sec. is only 1.2 per cent, as shown by Neher<sup>4</sup>. For the resolving time of the relay, which is also 0.02 sec., the percentage error is only one half per cent.

*The Transmitter*—The transmitters used in these flights were designed and made by Mr. A. S. Rao. Since the counting rate expected in these flights would be very low, the loss of a few counts would seriously affect the statistical accuracy of the observations. It was, therefore, decided to use a more powerful carrier than generally used in radio-meteorography. The choice of the wavelength was also made to make the man-made interference negligible. 144-150 mc. band was, therefore, chosen. Ordinarily used battery valves such as type 1G4, 19, etc., were found to be very poor at these wavelengths. The 958 type triode of 1.5



filament volt rating as used by Pickering<sup>8</sup> was not obtainable here and, therefore, a type 6C4, U.H.F. miniature triode, having 6.3 volt filament was chosen. Using an ultra-audion circuit as given in *A.R.R.L.* handbook<sup>9</sup>, with an input of 15 to 20 ma. at 270 volts on the anode, an R.F. output of approximately 1.5 watts was obtained in the half-wave antenna, capacitively coupled. With this carrier output, the signal strength during the flights was found to be good. In fact, in one of the trial flights, strong signals were obtained even at an altitude of 100,000'. There was not a single count missed due to fading in any of the cosmic ray flights, though, once or twice, data were lost while retuning the receiver to the carrier which drifted slightly. Fig. 3 shows the circuit of the transmitter and Fig. 4 is a photograph.

*The Meteorograph*—To determine the pressure corresponding to the position of the apparatus, at any instant during its flight, a meteorograph was needed. It was also necessary to know the temperature of the apparatus to know whether the change of temperature, if any, was such as to affect the counters, batteries and other equipment, sensitive to temperature.

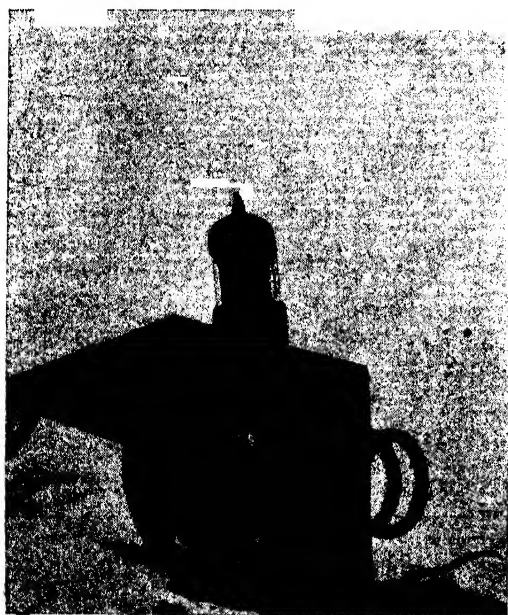


FIG. 4—THE TRANSMITTER, SHOWING THE TRANSMITTER ASSEMBLED ON A L-SHAPED CHASSIS. IT IS MOUNTED ON THE TELESCOPE FRAME DURING THE FLIGHT.

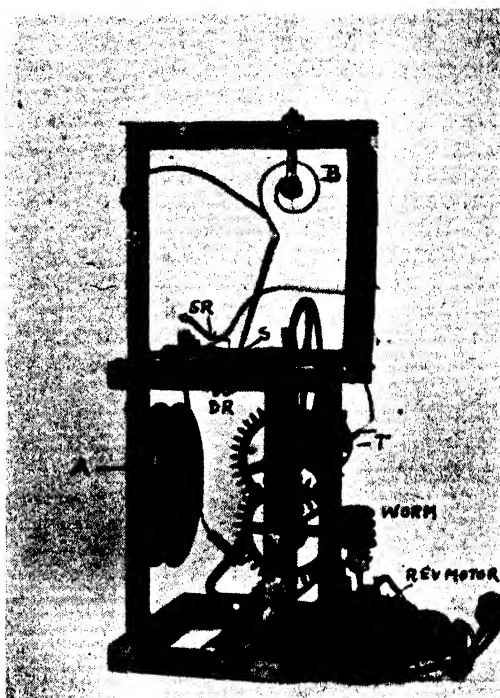


FIG. 5—THE METEOROGRAPH WITH MOTOR DRIVE—THE SMALL REV MOTOR IS SEEN COUPLED BY A 36:1 WORM.

A—Aneroid. B—Bimetal thermometer. S—Silver wire spiral. SR—Single reference for temperature. DR—Double reference for pressure. T—Ten teeth wheel with spring contact.

The coincidence unit, the transmitter, the H.T. and L.T. power supplies were enclosed in a bamboo cage, the whole of which was then enclosed in a double covering of violet and clear cellophane. The temperature regulation achieved by this will be described elsewhere.

A suitable meteorograph was available in the *India Meteorological Department*<sup>7</sup>. The original meteorograph is driven by a fan, operated by the downward wind as the meteorograph ascends with the balloon. Since, in these flights, the apparatus was enclosed, there was no air to drive the fan. The fan was, therefore, replaced by a midget motor operating on 4 to 6 volts battery. (These motors are manufactured by *Messrs Rev Motors Ltd.*, Bolton, England, and the radically new principle on which they work is described<sup>8</sup>.) The motor is only 1" in diameter and 0.5" long, and weighs only 22 gm. At 4.5 volts, it runs at 4,000 to 5,000 r.p.m. and the torque, after 36:1 worm

reduction, was found to be adequate to drive the meteorograph. Fig. 5 shows the meteorograph adapted for the motor drive.

The principle on which this meteorograph works is the same as in Bureau's<sup>9</sup> meteorograph. The working of the I.M.D. meteorograph and the method of evaluating the pressure-temperature data have already been described<sup>10</sup>.

Because of the design of this meteorograph, it was not possible to utilize the same carrier to transmit the cosmic ray and the meteorological data simultaneously as done by Neher and Pickering<sup>5</sup>. A separate transmitter was, therefore, used for the meteorograph channel similar to that used for the cosmic ray channel, but on a wavelength separated by a few megacycles. The meteorograph transmitter worked near 141 mc. and the cosmic ray transmitter near 151 mc.

It was, however, found that with such small separation between the two carrier frequencies, the receiver could discriminate between the two from the point of view of the receiver selectivity when the two transmitters were separated from each other at some distance. When both the transmitters were put in the same enclosure, the two resonating, half-wave aerials of very nearly equal lengths induced strong currents mutually, resulting in cross modulation. Since it was not possible to do radical design changes, either in the transmitter or the receivers, at the field station where the difficulty was observed without upsetting the time schedule of the flights, the only remedy was to separate the two transmitters to a minimum distance where the cross modulation was zero. The cosmic ray telescope, its transmitter and the necessary power supplies were, therefore, put in an enclosure covered with cellophane. The meteorograph, its transmitter and power supply were then put in another similar enclosure, and during the ascent it was hung 25' below the cosmic ray enclosure. Fig. 6 shows the two enclosures ready for ascent on the ground, and Fig. 7 shows the apparatus immediately after the release with the two enclosures hung one below the other.

*The H.T., L.T. & the Counter H.T. Power Supplies*—The apparatus was intended to work for 3 to 4 hours' maximum during the flights. Small, light-weight H.T. and L.T. batteries of adequately small capacities were not available to us. The smallest 1.5 volt cell commercially available



FIG. 6 — THE TWO ENCLOSURES READY FOR ASCENT. THE COSMIC RAY ENCLOSURE, BECAUSE OF ITS HEAVINESS, IS HUNG IN A PIPE FRAME TO FACILITATE THE ASSEMBLY OF THE VARIOUS UNITS IN THE BAMBOO CAGE. THE MAN ON THE RIGHT IS HOLDING THE METEOROGRAPH ENCLOSURE.

Photo by courtesy of Dr. L. S. Mathur

was the *Eveready* pencil torch, type 915, weighing 15 gm. each. H.T. and G.B. blocks were, therefore, made using these unit cells for the coincidence circuit and the transmitters. The capacity of these was found to be more than adequate for the current drain of these units, but the weight was excessive. The current drains of the various circuits were as under.

TABLE I

UNIT	H.T.		G.B. Volts	L.T.	
	Volts	Amps.		Volts	Amps.
Coincidence	90	0.003	36	1.5	0.3
Transmitter	270	0.02	...	7.5	0.180
Meteorograph motor	...	...	...	4.5	0.400

The L.T. units were made out of *Eveready* type 950 cells, two or more being connected in parallel to meet the current drain

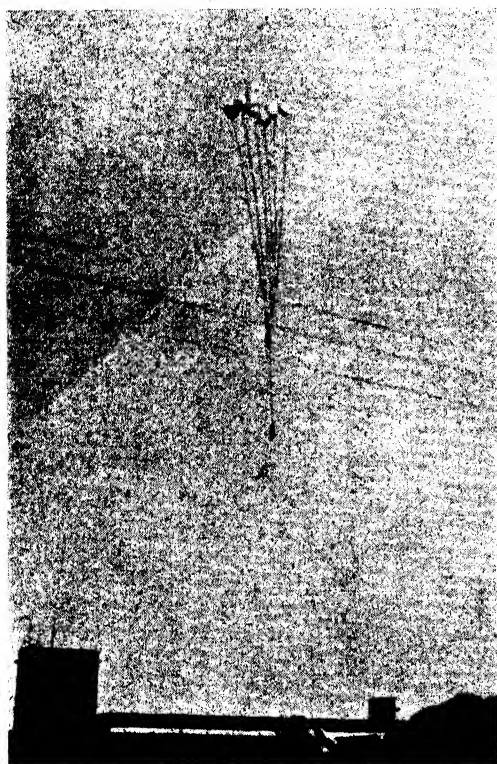


FIG. 7 — PHOTO SHOWS A FLIGHT AFTER RELEASE, WITH A CLUSTER OF EIGHT BALLOONS. THE TWO ENCLOSURES ARE CLEARLY VISIBLE HANGING BELOW THE BALLOONS. THE BOTTOM IS THE METEOROGRAPH ENCLOSURE.

Photo by courtesy of Dr. L. S. Mathur

requirements. Table II gives the total weights of the prepared H.T. and L.T. batteries.

TABLE II

TYPE		WEIGHTS	
		lb. oz.	gm.
Coincidence	H.T.	2 3	922.23
	L.T.	0 10	283.54
	G.B.	0 14	306.89
Transmitter	H.T.	7 2	3,231.83
	L.T.	2 3	992.23
Motor		1 4	566.99

*The Geiger Counter H.T. Supply* — Light-weight H.T. batteries were also not available commercially to make the 1,200-volt H.T. supply needed for the G-M counters. However, a technique was developed for preparing light-weight H.T. batteries for radio-meteorography<sup>10</sup> while the author was employed in the radio-meteorograph

development project of the *India Meteorological Department*. The technique was adopted for developing a suitable G-M H.T. supply. Smaller carbon cups similar to *I.M.D.* H.T. battery were supplied to us by *Messrs Helium Products Ltd.*, Poona. These cups were 0.5" in diameter and  $\frac{7}{32}$  high. A unit cell, after filling these cups with the depolarizing mixture, also supplied by the company, and putting a blotting paper disc saturated with electrolyte, on which a zinc disc was placed, gave 1.5 volts and weighed 1.6 gm. An adequate number of these cells were assembled in a "Volta Pile" fashion in a bakelite box having suitable compartments to give 1,200 to 1,300 volts H.T. as required. Since the current drain from the four counters on this H.T. was only a few micro-amperes, the only load on this battery was the leakage current in the assembled box. The battery was found to keep its terminal voltage within the range of the counter plateau for 2 hours after the complete battery was assembled. The assembly of these small cells, though simple, was rather tedious, and required about 10.5 man-hours. The assembly time was, therefore, brought down to 1.5 hours with a team of six to seven people. The complete H.T. battery after assembly weighed 6 lb. 4 oz. (2,835 gm.).

*The Weight of the Apparatus* — As mentioned above, the heaviness of the battery blocks contributed to the total weight of our equipment. Table III gives the weight of the different units in the balloon-borne equipment.

TABLE III

UNIT	COSMIC RAY ENCLOSURE		METEOROGRAPH ENCLOSURE	
	lb. oz.	gm.	lb. oz.	gm.
Bamboo cage	4 0	1,814.37	3 8	1,587.56
Cellophane bag	1 8	680.39	1 8	680.39
Coincidence unit with transmitter	4 0	1,814.37	...	...
Meteorograph with transmitter	...	...	2 6	1,077.28
Batteries for the transmitter	9 5	4,224.08	9 5	4,224.06
Batteries for the coincidence unit	3 11	1,672.61	...	...
TOTAL	22 8	10,205.82	16 11	7,569.20

Gross weight of the whole equipment: 48 lb. 2 oz. (21,857.37 gm.) with suspension rigging.

These weights are somewhat unconventional. Yet, using 10 to 15 balloons with 3,000 to 3,500 gm. free lift each, heights of about 40,000' were consistently reached;

in a few instances the flights reached up to 60,000'. In one flight, 10 cm. of lead was interposed between the counters of the telescope to measure the hard component of the cosmic ray intensity. The gross weight of the equipment in this flight was 64 lb. 6 oz. (29.2 kg.). This flight reached a height of 60,000', though the cosmic ray data were lost after 24,000', when the cosmic ray carrier drifted in frequency beyond the tuning range of the receiver.

### The Balloons

Latex balloons, manufactured by the *Nagpur Rubber Industries*, were used for these flights. Their NR-220 type, which is equivalent to *Dewey Almy Chemical Co.'s* (U.S.A.) neoprene balloon, *Darex J-800*, was used throughout. In previous ascents their performance was found to be comparable to the American equivalent. Like the American neoprene balloons, their life period for an afternoon flight was found to be 1.5 to 2 hours, irrespective of the rate of ascent or the height reached. The experience in these flights shows that it is desirable to use as few balloons as possible with the maximum permissible free lift to each, since the larger the number used, the

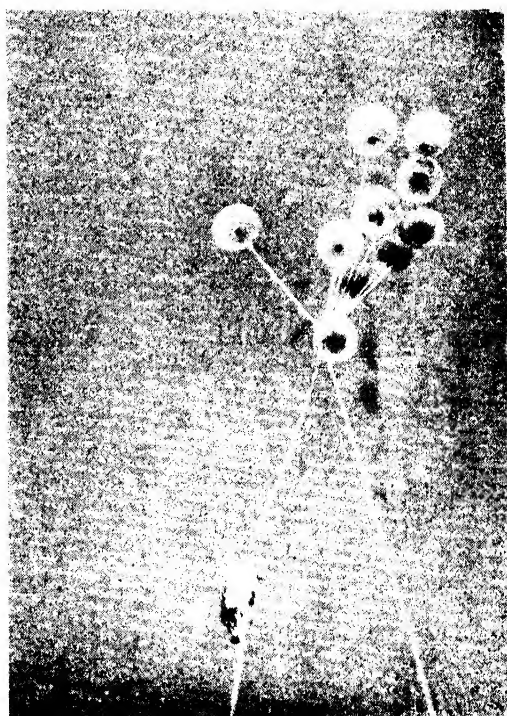


FIG. 8a — A CLUSTER OF TEN BALLOONS USED IN ONE OF THE FLIGHTS. THE CELLOPHANE WRAPPED COINCIDENCE ENCLOSURE IS ALSO VISIBLE.

*Photo by courtesy of Dr. L. S. Mathur*



FIG. 8 — SHOWS INDIAN MADE LATEX BALLOONS USED IN THESE FLIGHTS. NOTE THE PERFECT SPHERICAL SHAPE OF THESE BALLOONS.

*Photo by courtesy of Dr. L. S. Mathur*

greater is the probability of one or two bursting, limiting the maximum height reached. As many as 15 balloons were used in one of the flights with a smaller free lift than the maximum permissible lift when only 10 balloons were used. As mentioned before, heights of 40,000' have been consistently reached.

Figs. 8 and 8a show the balloons after release.

### The Ground Equipment

*The Receiver* — Neher and Pickering have used in their later balloon flights a super-heterodyne receiver for these U.H.F. carriers. This choice was made because, for the counting rate encountered in their experiments, the transmitter pulse was only a few micro-seconds long<sup>3, 11</sup>. Because of its inherent a.v.c. action, a super-regenerative receiver is unable to respond to such sharp pulses. In our flights the transmitted pulse was 0.05 second long, and hence could be recorded by a super-regenerative receiver. The additional advantage of the super-regenerative

receiver is that it does not respond to auto-ignition interference, and hence the choice of the receiving site is not as critical as when a superhet is used. Further, the meteorograph used requires a D.C. amplifier coupled to the recorder. This requirement is easily met by a super-regenerative receiver, since the hiss quenching of the receiver can be converted into a D.C. pulse of the same duration as the carrier pulse, which can be further amplified by a D.C. amplifier before being fed to the recorder.

Two super-regenerative receivers were, therefore, chosen to receive the two carriers from the cosmic ray equipment and the meteorograph. Commercially built *Mac-Murdo Silver Model 800* receivers were chosen. These receivers have only two tuning ranges of 144 to 148 mc./s. and 235 to 240 mc./s. The 144 to 148 mc. bands in the two receivers were slightly modified to cover 141 mc. and 151 mc. bands for our two channels. Because of this short tuning range and the broad tuning feature inherent in the super-regeneration principle and the design of the receivers, it was rather difficult to adjust our carrier sharply in the centre of the receiver band when the signal strength of the transmitter was very strong, although the transmitter was far away from the receiving location. Because of this difficulty, in one flight the cosmic ray carrier drifted beyond the tuning range of the corresponding receiver after the flight reached 24,000' although the maximum height reached by the flight was 60,000', as indicated by the pressure-temperature carrier, which was recorded up to this height.

*The D.C. Amplifier* — The D.C. amplifier used in conjunction with the above receivers is of the neon-coupled type<sup>10</sup>. Since the carrier for the cosmic ray channel is continuously on, except when a count is transmitted, the super-regeneration hiss in the corresponding receiver is normally quenched, except for the duration of the pulse. For the desired sensitivity, the number of stages in the neon-coupled amplifier is so chosen that, with the hiss quenched, the final stage in the amplifier is non-conducting. The recorder in its anode circuit is, therefore, normally not excited, and the pen attached to it is undeflected. When a pulse arrives, the hiss of the receiver comes on and the recorder marks a pulse. Fig. 9 is a circuit diagram giving the circuit of the super-regenerative receiver, the neon-coupled D.C. amplifier, and the voltage-regulated power

supply furnishing the H.T. and L.T. voltages, both for the receiver and the amplifier.

The complete system, therefore, consists of two such receivers coupled to D.C. amplifiers feeding the recorders with their separate power supplies.

*The Recorder* — The recorder is made out of a G.P.O., high speed, S.P.D.T. relay, with the coil rewound for lower impedance to work in the anode circuit of triode-connected 6F6 pentode in the final stage of the D.C. amplifier of Fig. 9. The relay contacts are removed to reduce the load on the armature and an extension is fitted to the armature arm, the other end of which carries a light-weight holder in which a "crow-quill" nib can be fitted. Two of such recorders are fixed opposite each other and write on a paper tape which is pulled by a tape-pulling arrangement made out of a variable speed, governor-controlled, gramophone induction motor, with a 9:1 reduction from its 78 r.p.m. spindle. The whole recorder is assembled on the front panel of the D.C. amplifier. The paper-tape stock spool, the idling rollers, the two recording pens with their vertical and lateral adjustments and the variable speed control knob of the tape puller drive, are thus always under view. Any adjustment of the recording nibs, their pen pressures, the ink feed, and the adjustments on the paper tape, if any fouling occurs, can thus be immediately made without stopping the recording, and thus no data are lost. The nibs of the recording pens are fed by cotton wicks from a small ink reservoir by capillary action.

Though the cosmic ray counts are continuously recorded, the pressure and temperature data are recorded once in one Olland cycle, the duration of which is from 2 to 4 minutes. To be able to interpolate the cosmic ray data with pressure corresponding to chosen height intervals, a continuous time trace is also put on the paper tape by a third pen. To reduce the complication of the ink-feed problem for a third pen, an ink refill, complete with the ball point, commercially available for an American made ball-point pen, was used for the third pen. This pen puts a continuous trace between the cosmic ray and the meteorograph traces. The time pen is lifted for a short duration every 15 seconds by an additional relay, similar to the recording relays, by the help of a synchronous clock. At every full minute there is a double interruption.

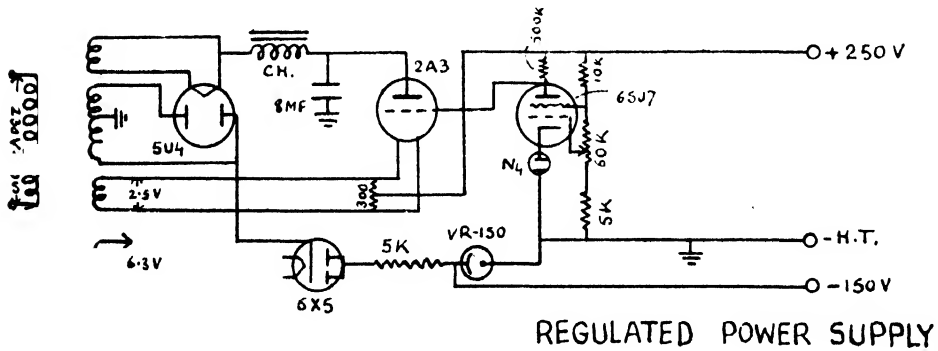
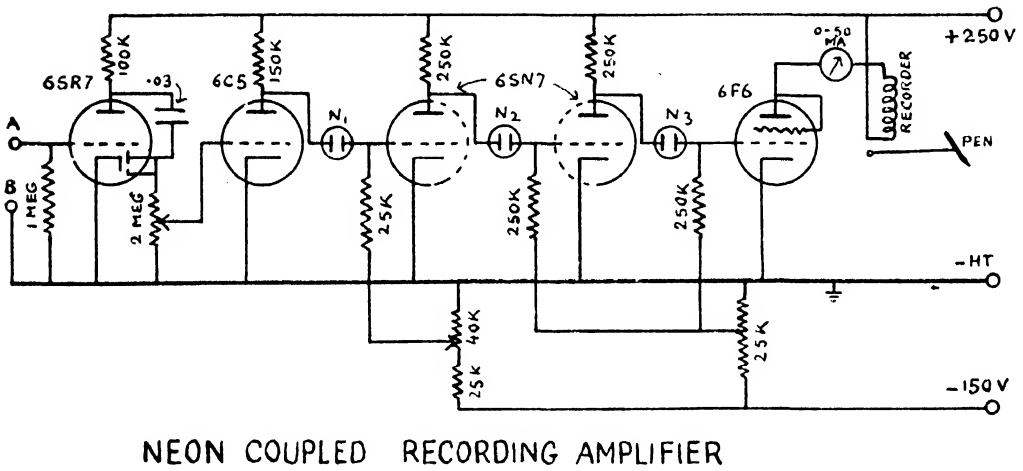
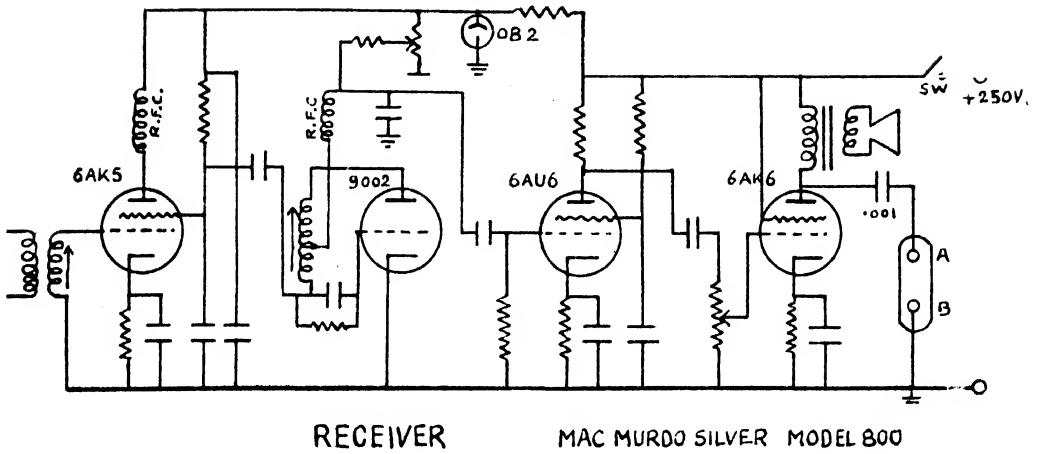


FIG. 9 — THE CIRCUIT OF THE U.H.F. SUPER-REGENERATIVE RECEIVER, THE NEON-COUPLED D.C. AMPLIFIER AND THE VOLTAGE-REGULATED POWER SUPPLY. TWO SUCH IDENTICAL UNITS WERE USED.



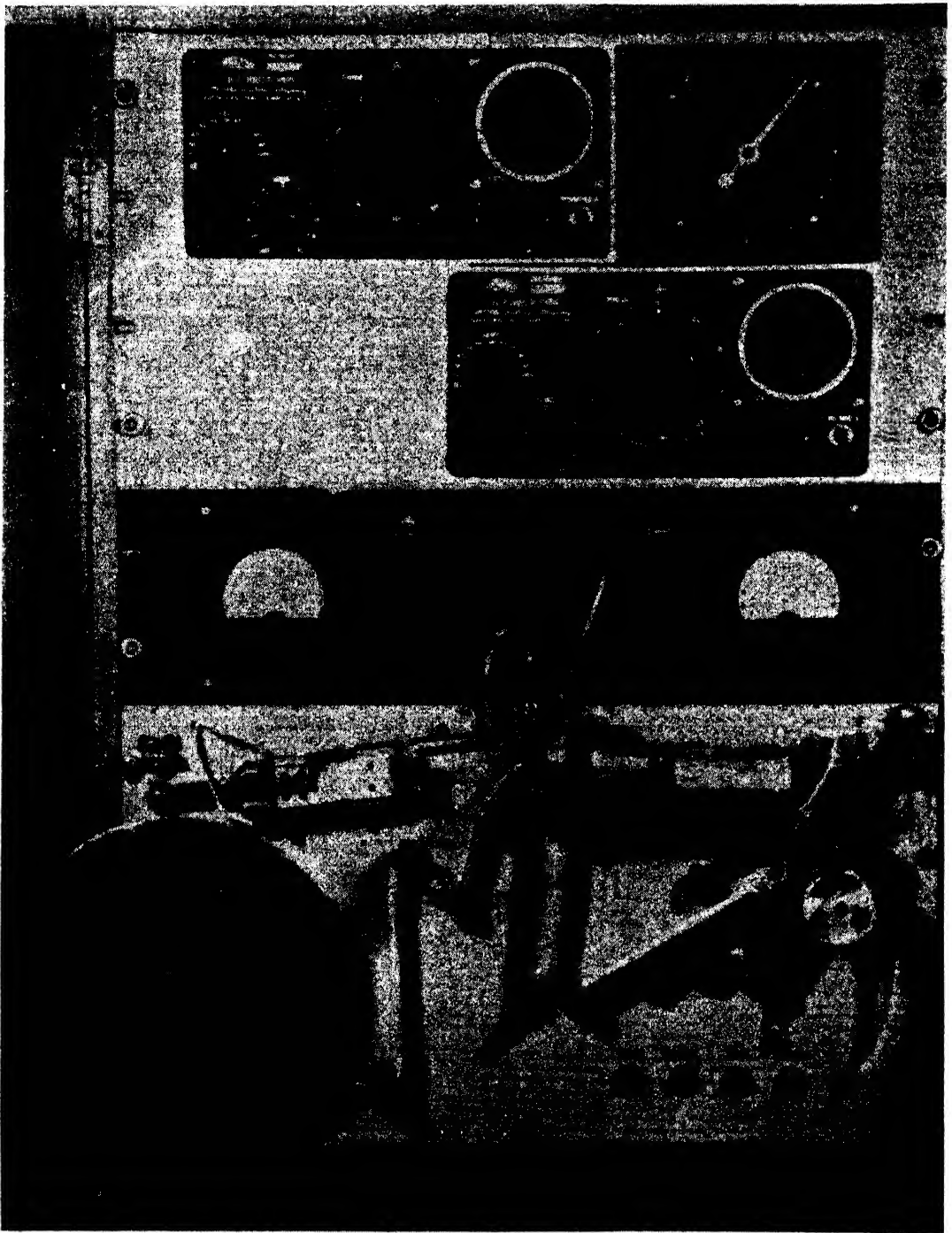


FIG. 10 — THE COMPLETE RECEIVING UNIT — THE TOP PANEL SHOWS THE TWO RECEIVERS AND THE TIME MARKING CLOCK. THE BLACK PANEL SHOWS THE TWO TUNING METERS AND THE SIX NEONS VISIBLE THROUGH CIRCULAR WINDOWS. ON THE BOTTOM PANEL ARE MOUNTED THE WRITING RELAYS AND THE TAPE-FEEDING AND PULLING ARRANGEMENT.

Thus, there is a continuous time recording synoptic with the two data records, and an accurate measurement of time to 0.01 minute can be made. From the pressure data a time-pressure curve is drawn. Having chosen a pressure interval corresponding to the desired height interval, the cosmic ray counts are measured from the tape for the corresponding time interval and the counting rate determined.

Fig. 10 shows the complete receiving set, rack mounted in a cabinet. The top panel carries the two receivers. The time marking clock is also visible in the right top corner. The next panel carries the six neons of the two D.C. amplifiers visible through windows. The two meters are 0 to 50 ma. meters in series with the recorder coils. These meters also serve as tuning indicators for the receivers, since the recorder current is zero for a perfectly tuned carrier, the strength

of which is above the threshold for positive operation of the recorder. The neon bulbs of the amplifier, which are visible, also help to keep a check on the satisfactory operation of the various stages of the D.C. amplifiers. The bottom panel shows the recording relays with the pens attached, the ink feed, the tape feed and the pulling arrangement. At the bottom is a small drawer carrying the accessories required for the recorder.

Fig. 11 is a photograph of a length of tape recording. The top trace shows the random cosmic ray counts, the middle is the time trace and the lowest is the meteorograph recording.

*The Receiving Aerial* — The receiving aerial is of the same type as designed by the author for radio-meteorography<sup>10</sup> in the *India Meteorological Department*. It has been found to be very successful in overcoming the periodic fading encountered because of

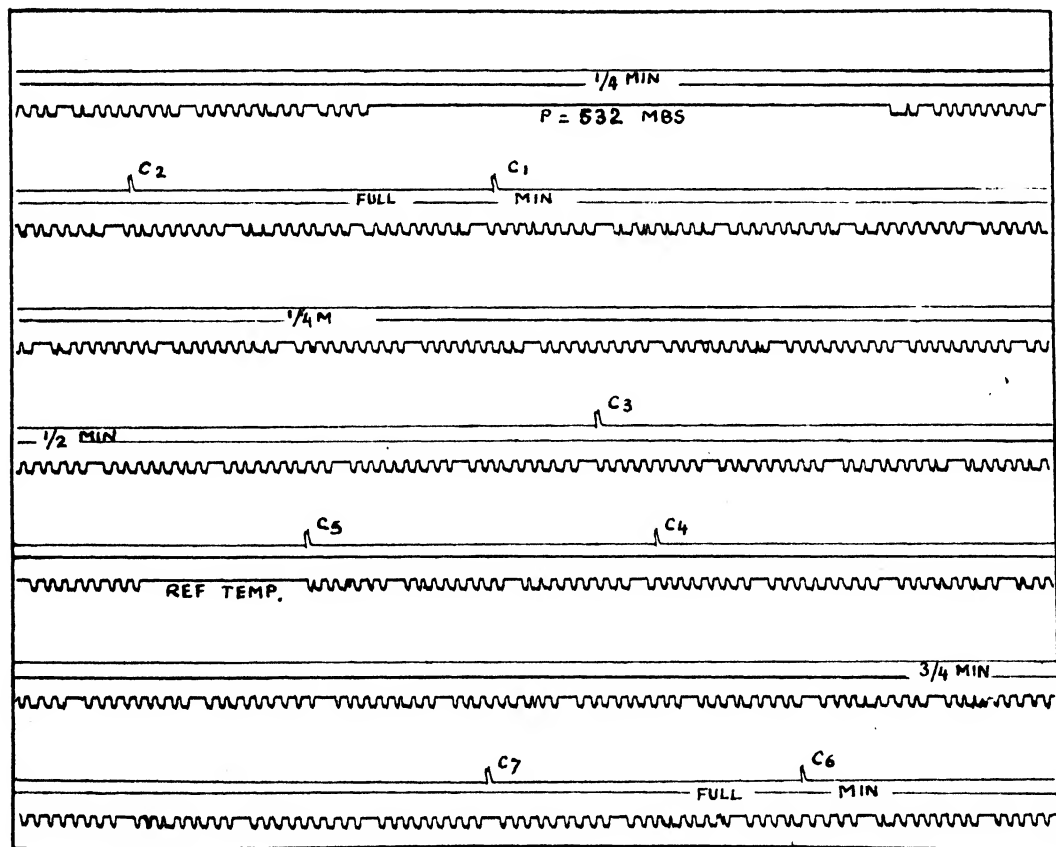


FIG. 11 — SAMPLE OF TAPE RECORD OBTAINED DURING ONE OF THE FLIGHTS AT ABOUT 5 KM. ALTITUDE. TOP TRACE IS FOR THE COINCIDENCE PULSES, THE MIDDLE FOR TIME, AND THE BOTTOM FOR METEOROGRAPH. THE DOUBLE INTERRUPTION AT EVERY FULL MINUTE AND SINGLE AT EVERY QUARTER MINUTE IS SEEN. SIX COINCIDENCE PULSES HAVE BEEN RECORDED FOR A ONE MINUTE INTERVAL.



swinging vertical antenna of the balloon-borne transmitter, which results in a change of the carrier polarization from the vertical.

### Summary

Using radio sonde technique, cosmic ray vertical intensity up to 40,000' was recently measured at New Delhi. The radio sonde equipment used in these flights is described. It consists of the coincidence unit with a pulse lengthening circuit; a meteorograph for the pressure-temperature data; and two U.H.F. light-weight transmitters to transmit the two data separately. The receiving unit consists of a super-regenerative receiver coupled to a neon-coupled amplifier feeding a high-speed tape recorder for each of the channels. Using Indian made balloons, heights of 40,000' were consistently reached, though the weight of the equipment was about 22 kg. In one flight, 10 cm. lead absorber was used, the total weight then being 30 kg., yet the flight reached a height of 60,000'.

### Acknowledgement

I would like to record my thanks to Messrs A. S. Rao and G. S. Gokhale in collaboration with whom the experiment was carried out. As mentioned in the body of the paper, the transmitters were made by Mr. A. S.

Rao who also duplicated the coincidence circuits. I also take the opportunity to thank Dr. Roy and Mr. S. P. Venkiteswaran of the *India Meteorological Department* for the use and supply of the Fan type meteorographs. Thanks are also due to Mr. Kalyansundaram of the *I.M.D's* Poona office, and Mr. Gokhale of this *Institute* for calibrating the meteorographs for these flights. I also wish to acknowledge with gratitude the encouragement received from Professor Bhaba during the course of this work. Finally, thanks are due to the *Council of Scientific & Industrial Research* for financial assistance for the scheme.

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## Micro-filming of Manuscripts

THE *Indian Library Association* IS CONSIDERING steps to be taken for filming perishing and perishable manuscripts and out-of-print books in India's libraries. The UNESCO is also interested in this work. It may be necessary, it is stated, to approach the UNESCO to provide micro-film laboratories in India in

the light of information regarding the micro-filming resources at the disposal of Government departments, research institutions and libraries in India. Such information about existing apparatus may be sent to Dr. S. R. Ranganathan, President, *Indian Library Association*, Delhi University, Delhi 2.

# Some Scientific Aspects of High-Polymers<sup>\*</sup>

LOURDU M. YEDDANAPALLI

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NATURAL products such as cellulose (in cotton, wood and vegetable fibres), protein (in silk and wool), and rubber (in *Hevea* latex) have, for a long time, been recognized as high-polymeric substances, that is, compounds made up of many (poly) identical, simpler units (mer) linked together by primary (covalent or homopolar) bonds. In cellulose molecule, the repeating unit (mer) is anhydrous glucose ( $C_6H_{10}O_5$ ); in silk-fibroin, glycylalanine residue ( $--NH-CH_2--CONH-CH(CH_3)-CO--$ ), and in rubber, isoprene ( $-CH_2-C(CH_3)=CH-CH_2-$ ). In recent years, a large number of synthetic high-polymers with valuable properties and of great practical utility has come into existence in the shape of textile fibres (rayons, nylons, vinyons, etc.), rubbers (Buna S, Buna N, Butyl, Neoprene, etc.), and plastics of a bewildering variety. Scientific research carried out particularly during the last 20 or 25 years, both on natural and synthetic high-polymers, has brought to light certain fundamental characteristics common to all these substances and also some specific differences which are responsible for their division into fibres, plastics and rubbers. A knowledge of these similarities and differences, a critical survey of which is proposed to be given in the present article, is necessary not only to understand

the nature of high-polymeric substances, but also to be able to synthesize new materials possessing better and more useful properties; in short, to synthesize the so-called tailor-made molecules.

## Molecular Weight

The first characteristic of all high-polymers is their molecular weight. Classical organic chemistry deals with substances whose molecular weights rarely exceed a thousand. The molecular weights of high-polymers run into tens and hundreds of thousands as may be seen from Table I. They have been determined by osmotic pressure<sup>2</sup>, viscosity<sup>3</sup>, ultra-centrifuge<sup>4</sup> and, more recently, light-scattering<sup>5</sup> methods. But there is a certain indefiniteness about them. When we speak of sugar having a molecular weight, 342, we understand that each sugar molecule in a given sample has the same number of carbon, hydrogen and oxygen atoms, and hence has the same size and shape. But, in the case of a high-polymer, all the molecules in a given sample do not appear to have the same number of fundamental units, nor the same size and shape; some are longer, some more branched than others. In fact, there seems to be a regular distribution of size and shape among the molecules of a given sample so that we can speak only of an average molecular weight.

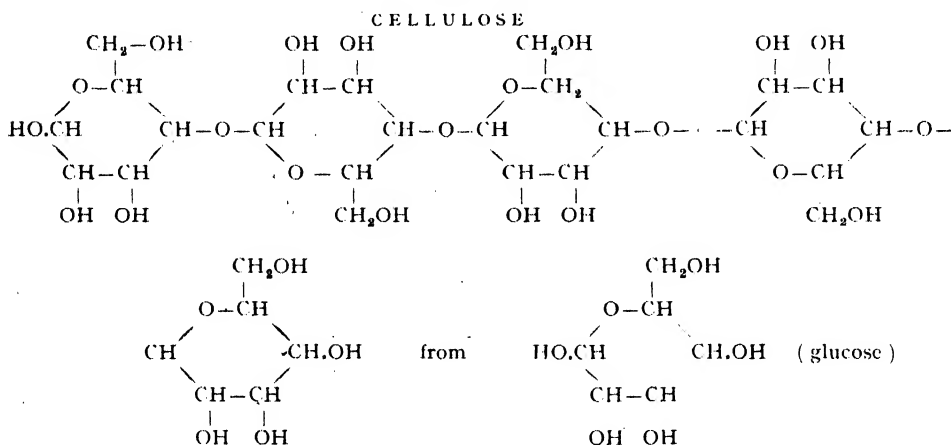
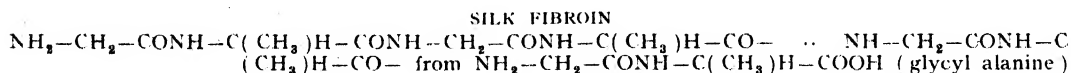
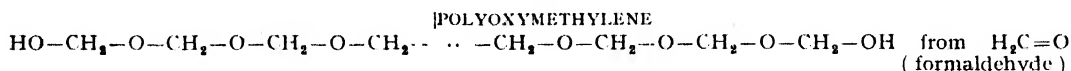
TABLE I — MOLECULAR WEIGHTS & DEGREE OF POLYMERIZATION (D.P.) OF SOME HIGH-POLYMERS

MATERIAL INVESTIGATED	MOL. WT.	D.P.
Native cellulose in cotton, ramie, or wood	300,000—500,000	2,000—3,000
Cellulose in bleached cotton linters	150,000—230,000	1,000—1,500
Cellulose in purified wood pulp	120,000—200,000	800—1,200
Regenerated cellulose in rayon	75,000—100,000	500—600
Regenerated cellulose in staple fibre	60,000—75,000	400—500
Regenerated cellulose in cellophane	50,000—60,000	300—400
Native rubber in <i>Hevea</i> latex	140,000—210,000	2,000—3,000
Rubber after being milled in air	55,000—70,000	800—1,000
Nitrocellulose used for moulding	400,000—700,000	1,500—2,500
Nitrocellulose used for extrusion	150,000—300,000	600—1,200
Nitrocellulose used for coatings	50,000—100,000	200—400
Polystyrene for plate casting	250,000—400,000	2,500—4,000
Polystyrene for injection moulding	120,000—180,000	1,200—1,800
Polystyrene for coatings	80,000—120,000	800—1,200
Polyvinyl chloride (Koroseal)	250,000	3,000
Polyisobutylene (Vistanex)	120,000—200,000	2,000—3,000
Polyhexamethylene adipamate (Nylon)	16,000—32,000	150—300

<sup>\*</sup> Based on a paper read at the High-Polymer Symposium, Chemistry Section, Allahabad Session of the Indian Science Congress Association, 1949.

Experimental justification for this statement has amply been provided by fractional precipitation, first of rubber by Caspari<sup>6</sup>, later of nitro-cellulose by Duclaux and Wollman<sup>7</sup>, and by the work done on cellulose balta and many synthetic polymers by Staudinger and coworkers<sup>8</sup> from 1927

as cellulose, silk-fibroin, polypeptides, polyoxymethylene, polyethylene glycol, in which, though the repeating unit (mer) in a particular high-polymer is the same, the chemical composition of the latter is not identical with that of the starting simple molecule, as illustrated in the following structures:



onwards, and on rubber by Midgley and coworkers<sup>8</sup> from 1931. The influence of molecular weight distribution curve on the mechanical properties of high-polymers will be discussed later.

### Polymerization

Whatever be the manner in which high-polymeric materials are synthesized by nature in plant and animal organisms, the process by which they have been produced from simpler molecules in the laboratory or in industry is known as polymerization. Classical organic chemistry, interpreting Berzelius' definition of a polymer in a rather narrow sense, restricted the capacity to polymerize to molecules containing an unsaturated bond, such as  $-\text{C}=\text{C}-$ ,  $-\text{C}=\text{O}-$ ,  $-\text{C}=\text{N}-$ , so that the polymer would have the same chemical composition as the simple molecule from which it was built up. The inadequacy of such a definition of a polymer and of polymerization was first pointed out by W. H. Carothers<sup>9</sup> from a critical examination of the structures of such admittedly high-polymeric substances

He showed from these examples that the characteristic feature of a high-polymer is the structural unit, the repetition of which leads to the large molecule. He proposed, therefore, as a more logical and comprehensive definition of polymerization any chemical union of similar molecules to form a single large, i.e. high-polymeric molecule. Such a chemical combination may be effected either by mere self-addition, or by condensation involving the elimination of simple molecules such as water, ammonia, hydrochloric acid, etc., so that one can speak of addition or A-polymerization, and condensation or C-polymerization.

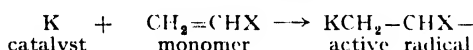
Addition polymerization<sup>10</sup> is undergone essentially by molecules containing an unsaturated linkage. Thus, ethylene can be polymerized into the technically valuable polyethylene<sup>11</sup>; known for its excellent insulating properties:

$\text{CH}_2=\text{CH}_2 + \text{CH}_2=\text{CH}_2 \rightarrow -\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-$   
which, with further addition of ethylene molecules, results in a large molecule. But the reaction requires high temperature,

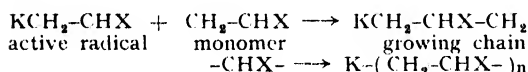
200° to 300°C. and high pressures up to 1,200 atm., though lower degrees of polymerization can be easily achieved<sup>12</sup>. On the other hand, substituted ethylenes, of the type  $\text{CH}_2 = \text{CHX}^{13}$ , where X is a phenyl or carboxylic ester group or a halogen atom, undergo polymerization much more readily. Examples are: polystyrene from styrene, polyvinyl acetate from vinyl acetate, polyacrylates from acrylic esters, polyvinyl and polyvinylidene chlorides from vinyl and vinylidene chlorides respectively.

This type of polymerization, also known as vinyl polymerization because of the compounds involved, is generally carried out with the help of a catalyst<sup>14</sup> such as free radicals, and free radical yielding organic peroxides (benzoyl peroxide, diazo-compounds, etc.), and metal halides like  $\text{BF}_3$ ,  $\text{AlCl}_3$ ,  $\text{SnCl}_4$ . The chemical process appears to be a chain reaction involving :

(a) activation of the monomer by a catalyst leading to an activated state having the character of a free radical.

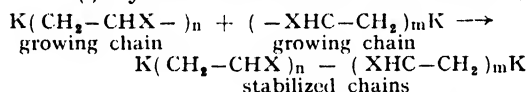


(b) followed by successive additions of monomer molecules.

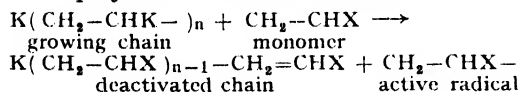


(c) until the polymer is stabilized either :

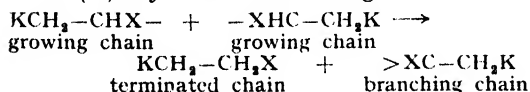
(i) by chain termination or cessation,



or (ii) by chain transfer in which the activity is transferred to some other polymer or solvent molecule,



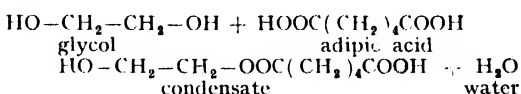
or (iii) by chain branching,



It appears that process (b), having a low activation energy, is frequently repeated, about a thousand times or more in a fraction of a second, so that the chain grows to a fair size in a comparatively short period of time before it is terminated by a cessation process. Consequently the average chain length (or degree of polymerization, D.P., indicating the number of monomeric units

in the chain) and consequently the average molecular weight will be independent of time or extent of the reaction, unlike in the case of the other type of polymerization, as will presently be seen.

**Condensation polymerization**<sup>9,15</sup> consists of a stepwise combination of the reactive groups of monomer molecules with the elimination of simple molecules like water, etc. Thus,



The OH and COOH groups of the resulting molecule can further react with COOH and OH groups of other monomeric molecules respectively to give, after successive condensations, a long chain molecule. Similarly diamines, such as hexamethylene diamine, can react with adipic acid to yield polyamides like nylon. The same type of polymerization is involved in the industrial preparation of plastic as well as varnish and adhesive resins from phenol and formaldehyde, urea or melamine and formaldehyde, glycerol and phthalic anhydride, and of polysulphide rubbers (Thiokol) from alkyl dihalides and alkali polysulphides, with the elimination of alkali halide molecules. Because of the stepwise character of the reaction, the average chain length (even if branched) and the average molecular weight of the resulting polymer (leaving aside for the moment cross-linked polymers) will depend on the extent of the reaction.

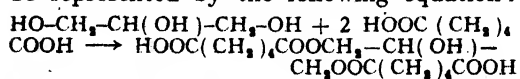
It may not be out of place to point out here a variation of the simple addition polymerization, known as copolymerization, first discovered by Klatte in 1914, now extensively employed in industrial practice, and still being actively investigated in some of its fundamental aspects<sup>16</sup>. It consists in the simultaneous polymerization of 2 or more vinyl compounds such as vinyl acetate and vinyl chloride to get a polyvinyl acetate chloride copolymer (vinylon). Buna S, a copolymer of butadiene and styrene, was successfully substituted for natural rubber on a large scale by U.S.A. during the last war. Buna N is a copolymer of butadiene and acrylonitrile and has excellent resistance to hydrocarbon and other organic solvents.\*

\* Regarding the manner of addition polymerization, bulk polymerization was the common practice until some years ago; now, emulsion, employed by U.S.A. for Buna S and more recently, suspension polymerization are becoming more important<sup>18</sup>.

### Shape of High-polymers, Linear & Cross-linked

The second characteristic of a high-polymer resulting from either of the above type of polymerization is the shape of its molecules which may be a straight, linear, or a cross-linked, 2 or 3-dimensional structure. There is, of course, the possibility of branched chains as is suggested by the mechanism of addition polymerization already described above. But, for the present discussion, a linear chain with only some degree of branching may be approximated to a straight linear type, and one with a high degree of branching may be regarded as equivalent to a cross-linked molecule. The production of these 2 widely differing molecular structures depends primarily on the number of reactive, functional groups in the monomeric molecule or molecules<sup>9</sup>. If the monomer is bifunctional, that is, has only 2 reactive groups or a single double bond, the polymer resulting from it by either type of polymerization will be essentially a linear chain as is clear from the examples already adduced. Polyesters from glycols and dicarboxylic acids, polyamides from similar acids and diamines, and even phenolic resins (Novolak) from *para* cresol with only two reactive hydrogen atoms in the *ortho*-positions to the phenolic OH and formaldehyde, will contain mainly linear molecules. That there is possibility of some degree of branching in the case of vinyl polymers is evident from the mechanism of the process already outlined and from indications supplied by viscosity measurements<sup>17</sup>.

If the monomers are polyfunctional, that is, having more than 2 reactive groups in each molecule, there is definite possibility of growth of the polymer in more than 2 directions in space leading to a complex net and 3-dimensional structures. Thus, if glycerol, instead of glycol, is condensed with adipic acid, the resulting compound may be represented by the following equation:



The secondary OH of glycerol can next react with the  $-\text{COOH}$  of another acid molecule, whereas the  $-\text{COOH}$  groups can react with OH groups of other glycerol molecules. As the reaction proceeds, further condensations not only in 2 but in 3 directions can take place to give finally a complex cross-linked, space polymer. Similarly,

glycerol and phthalic anhydride react to yield Alkyds or Glyptals used as varnish resins. Phenol, which has 3 reactive hydrogens in the nucleus, condenses with formaldehyde to give phenolic resins (Bakelite), urea with 4 reactive hydrogens and melamine with a still higher number of reactive hydrogen atoms combine with formaldehyde to yield complex polymers, ureas and melamines.

It is of interest to note that the above division of synthetic high-polymers into linear and cross-linked molecular structures corresponds to what obtains in natural high-polymers such as cellulose, chitin, rubber, which are mainly long-chain, thread-like molecules, and keratin (with S-S cross links), lignin, etc., which are cross-linked, complex structures.

### Physical & Chemical Properties

The physical and chemical properties of these 2 types of polymers are quite distinct and of technical importance<sup>19</sup>. The chain polymers have an indefinite capacity to swell in suitable solvents and eventually pass into solution in molecular state, since there are no strong chemical bonds between adjacent chains to hold them together. In the swollen state they are capable of undergoing exchange reactions such as esterification, chlorination, saponification, etc., without apparently changing their average size or shape. Thus polyvinyl acetate can be hydrolysed into the corresponding alcohol which in turn can be condensed with aldehydes to give polyvinyl acetals, as is done in industrial practice; polyvinyl butyral, for instance, being used as self-sealing inner lining of gasoline tanks of fighter planes. The temperature effect on chain polymers (of the plastic type) to distinguish them from the fibre type) is very characteristic and of technical value. With temperature, they pass from the amorphous or quasi-crystalline state through a softening or plastic zone into a viscous, liquid condition. This change with temperature is indefinitely reversible and is characteristic of what are called thermoplastic high-polymers. This behaviour with temperature has recently been explained by Mark<sup>20</sup> in terms of micro- (irregular motion of segments of chains) and macro- (displacement of chains as a whole) Brownian movements, originally introduced by Kuhn. Only thermoplastic materials can be utilized for the rapid methods of fabrication, such as injection

moulding and extrusion processes, in the place of the time-consuming compression moulding.

The net and space polymers, on the other hand, show only a limited capacity to swell in solvents. Because of the chemical cross-links between the chains, the material does not go into solution at all. Phenolics, ureas, certain polyesters are typical examples. They do not undergo permutoid reactions like the chain polymers and are, therefore, chemically more inert than the latter. They show no definite zone of softening with temperature and at relatively high temperatures they suffer chemical decomposition and are known as thermosetting plastics. Because of their thermosetting character, special procedure has to be employed in moulding operations. For instance, in the case of phenol-formaldehyde resins, the polymerization is first carried only to the thermoplastic stage (A and B stages) in which there are few cross-links between the long chains and, in the mould, along with fillers, plasticizers and catalyst, the material is made, under pressure and temperature, to react to the final thermosetting condition. A similar procedure is adopted in the case of urea resins, for otherwise fully reacted thermosetting resin is incapable of the plastic flow necessary to take the shape of the mould. The compactness of molecular structure of thermosetting plastics is responsible for their chemical inertness, electrical properties and resistance to mechanical abrasion and also for their brittle character. Plasticizers and fillers are, therefore, necessary to give them toughness and impact and tensile strength. A fully cured thermosetting plastic material is something like a giant molecule or an aggregate of such molecules, so that it is not possible to evaluate their molecular weight or exact shape and size. Hence the following discussion is limited to thermoplastic and, in general, to chain polymers.

#### Molecular Weight & Mechanical Strength

The dependence of mechanical strength of chain polymers on the average molecular weight or average chain length was first investigated by Rocha, Ohl and Carothers, and later by several authors, particularly, Harris, Sookne, Spurlin, Mark and Meyer. It appears<sup>21</sup> that for a high-polymer to begin to manifest any mechanical strength at all, it must have a certain critical chain length

or D.P. (around 40 for polyamides, and 80 for polyhydrocarbons) above which the strength increases with increasing D.P. up to a limiting value (around a D.P. of 600 to 700) beyond which further increase in chain length does not seem to affect sensibly the mechanical strength, as can be seen from Fig. 1 (after Mark)<sup>21</sup>. The upper curve, with circles, refers to polyamides and the lower curve, with crosses, to polyhydrocarbons. The minimum and maximum D.P. limits for polyamides are lower than the corresponding values for hydrocarbons, because of the additional contribution made to mechanical strength by the cohesive forces between the chains coming from the polar groups along the polyamide chains. When the chains are sufficiently long for their ends to be effectively entangled so as to avoid slipping under tension, tensile strength or tenacity will become independent of further increase in D.P. The shaded region between the two curves refers to all other high-polymers such as cellulose esters, polyvinyl derivatives, etc. (Figs. 1 and 2).

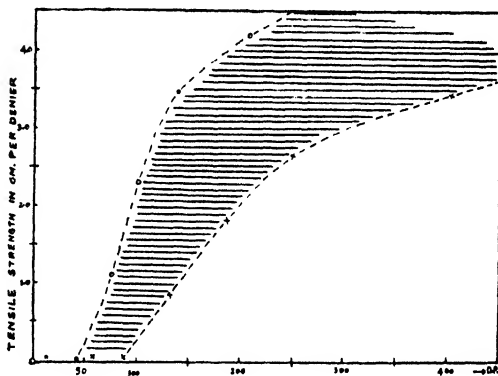


FIG. 1 — SCHEMATIC REPRESENTATION OF THE RELATION OF THE TENSILE STRENGTH WITH AVERAGE DEGREE OF POLYMERIZATION.

In this connection a less well-understood relation is the influence of the range of distribution of chain length or D.P. on the mechanical strength, in spite of the researches carried out by various authors, especially Mark, Harris, Sookne and Spurlin. According to Mark, "the shape of the distribution curve seems to have no effect on the ultimate strength except that a comparatively small amount (between 10 to 15 per cent by weight) of constituents having a polymerization degree below 150 is definitely detrimental to the mechanical properties of the

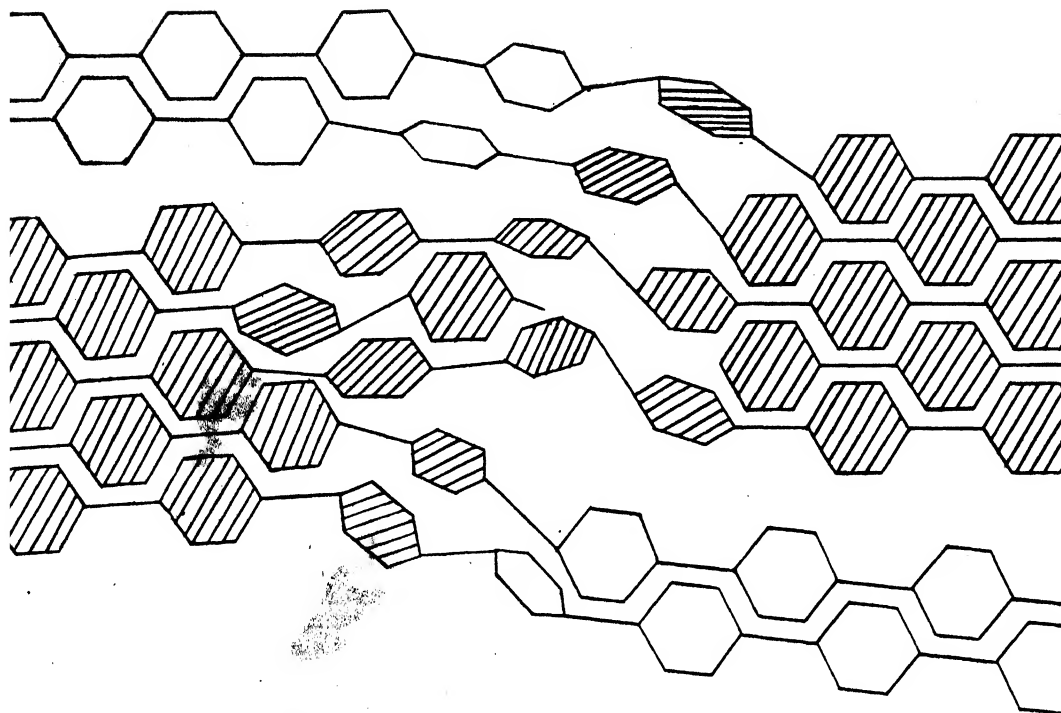


FIG. 2 — DIAGRAMMATIC REPRESENTATION OF CELLULOSE CHAINS WHICH ARE PARTLY BELONGING TO CRYSTALLITES AND PARTLY TO A DISORDERED AREA.

sample."<sup>22</sup> A more comprehensive statement based on the work of the other authors cited above is presented by Alfrey<sup>23</sup>, though it does not contradict the general conclusion of Mark. It may be added that in general the more uniform the chain lengths, provided the limiting D.P. is attained, the better would be the mechanical properties of the sample.

#### Molecular Texture & Mechanical Properties

It is the molecular architecture which is ultimately responsible for a high-polymeric material being a fibre, plastic or rubber. In the study of this molecular geometry, the X-ray method of Debye and Scherrer has been of particular value. Well-known organic crystals, in powder form, give an X-ray pattern known as powder diagram, consisting of bright spots of varying intensity and sharpness from which the elements of symmetry of the crystalline material can be evaluated. An amorphous material, on the other hand, yields an X-ray pattern of diffuse rings or halos, commonly known as diffuse ring diagram, revealing the absence of

crystalline order in the diffracting material. Natural rubber in its unstretched condition gives such a diffuse ring diagram as was first reported by Scherrer<sup>24</sup>. Cotton fibre<sup>25</sup> gives a diffraction pattern, known as fibre diagram, of a certain intensity and sharpness, intermediate between that of a regular crystal and of an amorphous material. This has been interpreted to mean that cotton fibre contains ordered regions of a certain degree of crystallinity (crystallites) which are separated from one another by disordered (amorphous) regions, the crystallites being so oriented that the molecules from which they are built are parallel to the fibre axis. Also there is no sudden, but only gradual, transition from the ordered to the disordered regions. One and the same chain is supposed to find itself partly in the crystalline and partly in the amorphous domains of the fibre, as illustrated in Fig. 2 (after Mark)<sup>26</sup>. An interesting discovery was first made by Katz<sup>27</sup> on rubber that, when stretched, it gives a fibre X-ray diagram indicating the presence of a certain degree of crystallinity brought about by the mechanical process of

stretching. In this stretched state, rubber acquires its tensile strength. It is natural, therefore, to associate tensile strength with crystalline structure both in stretched rubber and ordinary cotton fibre. The increased tensile strength of strong rayons (regenerated cellulose) is to be ascribed to the better alignment of chains in the crystalline regions induced by the mechanical stretching during spinning, as shown by an enhanced fibre diagram. R. O. Herzog<sup>28</sup> was one of the first to study the quantitative relationship between the tensile strength of viscose and cuprammonium rayons and the improved orientation of crystallites parallel to the fibre axis as revealed by X-ray diagrams. Further studies on the subject led to the common industrial practice of stretch-spinning and to the discovery by Carothers<sup>29</sup> of the cold-drawing process of nylon as well as, in recent years, of vinyon and other polyvinyl fibres and filaments.

An interesting feature to be noted about the cold-drawing of nylon and the other fibres is that the fibre retains, unlike rubber, the impressed elongation after the removal of the stretching force. Even in the unstretched condition, nylon, like natural proteins, is supposed to have its chains folded in some regular fashion, and on being stretched by external force, as the chains become parallelized, the polar side groups in the chains, CO and NH groups, by their hydrogen bonding, maintain the chains in the stretched state. As Mark has put it, in the case of nylon, internal crystallization is converted into external crystallization by the cold-drawing process. In the case of rubber, on the other hand, such strong polar group forces do not exist between the hydrocarbon chains. Further, the methyl groups arranged in *cis*-position along the chains tend naturally to bring the chains, once the external stretching force is removed, into the thermodynamically stable position of maximum entropy, which accounts for the elasticity of rubber. When unvulcanized rubber is subjected, however, to a prolonged tension or stretching, it begins to creep, that is, the chains tend to slip past one another, since there are no strong cohesive forces between the chains, and it does not snap back quickly. But vulcanized rubber does not suffer creep, and snaps back quite effectively, because vulcanization introduces, here and there along the chains, sulphur links at the place of the double bonds which prevent slippage of the chains and pull

back the chains to their natural curled-up state on the removal of the stretching force. It may be noted that if the sulphur cross-links between the chains are closely spaced by the incorporation, into rubber, of a large amount of sulphur, a rather compact molecular structure is obtained which is hard rubber or ebonite.

From the foregoing discussion, certain general conclusions<sup>1</sup> can be deduced regarding the relation between the mechanical properties and molecular structure of high-polymers. Once the required chain length and fair uniformity of chain length are secured, it is the nature of the chain structure and the cohesive forces between chains which determine whether the material is a typical fibre, plastic or a rubber. If the chains easily fit into some regular geometrical pattern and retain that pattern by virtue of strong intermolecular forces arising from the presence, along the chains, of dipole or polarizable groups or hydrogen bonds, the material will be a typical fibre. On the other hand, if the chains do not fit well into a regular geometrical pattern, either because of bulky side chains (as in the case of Buna S) or because of unsymmetrical disposition of side groups (as in the case of *cis* arrangement of methyl side groups in natural rubber), and if there are no strong cohesive forces between the chains, the material would behave as a typical rubber. Between these two extremes are to be found plastic materials with intermediate geometrical structures and intermolecular cohesive forces; given the possible combination of the relative magnitudes of these two factors, a great variety of plastics is possible. Table II (after Mark)<sup>30</sup> illustrates the type of cohesive forces between chain molecules of the 3 categories of high-polymers. The values for the cohesive forces, presented in the last column of the table, are in cal. per mol. for a chain length of 5 Angstrom units on the assumption that each chain is surrounded by 4 others, as suggested by the X-ray structure of these polymers. For rubbery materials the forces have an energy value around 1,000 cal. per mol., and for fibres, a value around 6,000 cal. That the intermolecular forces alone are not sufficient to explain the mechanical properties of the polymers is clearly indicated by some of the listed substances. Thus, polyethylene with only 1,000 cal. intermolecular forces tends to show rather fibre than rubbery properties, because, in spite of the weak



TABLE II—INTERMOLECULAR FORCES OF SOME HIGH-POLYMERS &amp; THEIR INFLUENCE ON MECHANICAL BEHAVIOUR

SUBSTANCE	COVALENT BOND ENERGY ALONG THE CHAINS, IN. CAL. PER MOLE	MOLAR COHESION PRO 5 Å CHAIN LENGTH WITH CO-ORDINATION NUMBER FOUR, IN CAL. PER MOLE
Polyethylene	-C-C- 70-80,000	(CH <sub>2</sub> ) 1,000
Polyisobutylene	-C-C- 70-80,000	(CH <sub>2</sub> ), (CH <sub>3</sub> ) 1,200
Polybutadiene	-C=C- 70-120,000	(CH <sub>2</sub> ), (CH=CH) 1,100
Rubber	-C=C- 70-120,000	(CH <sub>2</sub> ), (CH=C-CH <sub>3</sub> ) 1,300
Polystyrene	-C-C- 70-80,000	(CH <sub>2</sub> ), (C <sub>6</sub> H <sub>5</sub> ) 4,000
Polychloroprene	-C=C- 70-120,000	(CH <sub>2</sub> ), (CH=CCl) 1,600
Polyvinyl chloride	-C-C- 70-80,000	(CH <sub>2</sub> ), (CHCl) 2,600
Polyvinyl acetate	-C-C- 70-80,000	(CH <sub>2</sub> ), (COOCH <sub>3</sub> ) 3,200
Polyvinyl alcohol	-C-C- 70-80,000	(CH <sub>2</sub> ), (CHOH) 4,200
Cellulose	-C-O-C- 80-90,000	(H), (-O-) 6,200
Cellulose acetate	-C-O-C- 80-90,000	(OCOCH <sub>3</sub> ), (-O-) 4,800
Polyamide	-C-N-C- 70-90,000	(CH <sub>2</sub> ), (CONH) 5,800
Silk fibroin	-C-N-C- 70-90,000	(CH <sub>2</sub> ), (CONH) 9,800

forces, its chains, the simple methylene zigzag hydrocarbon chains, fit easily into a crystal lattice, and polyethylene is, therefore, a plastic with fibrous tendencies and, under proper mechanical treatment, can be made even into filaments. Nylon, with cohesive intermolecular forces less strong than those of silk, shows greater mechanical strength, because its chains fit into a crystal lattice better than the chains of silk fibroin, or even those of cellulose. Polystyrene and polyvinyl acetate with forces approaching those of a fibre still are typical plastics because of the bulky benzene rings in the one and the acetate groups in the other which do not facilitate an easy fitting of the chains into a crystal lattice. But if the intermolecular forces are weakened by the elimination of hydrogen bonds and the ease of crystallization of the chains reduced by the replacement of the hydrogen atoms of -NH- by methyl or methoxy groups, the resulting nylon will acquire elastic properties characteristic to rubber, as was reported by Baker and Fuller<sup>31</sup>, Briggs and others<sup>32</sup>, and recently by Hill and Walker<sup>33</sup>.

#### Influence of Side Groups

An interesting example of side group influence is that of polymethacrylate plastics<sup>34</sup>, in which the side chain groups bring about considerable modification of physical properties. Polymethyl-methacrylate is the well-known tough, elastic, celluloid-like Lucite (or Plexiglass or Perspex); polyethyl-methacrylate is less tough, softer and elastic; polybutyl-methacrylate is soft and sticky and finally polyoctyl-methacrylate is jelly-like, and the corresponding lauryl compound is a viscous liquid. With increasing length of side groups, the main long chain molecules are pushed apart more and more until the intermolecular forces become so weakened

that the chains acquire almost the freedom they would in the liquid state, hence the viscous liquid properties of the lauryl-methacrylate resin. It is of interest to examine here the solution of the problem of polymerizing isoprene to rubber. When isoprene is polymerized, the product is not the elastic natural rubber but a hard gutta-percha in which the methyl groups are mostly in the *trans* position, so that the chains fit well into a regular lattice and lead to the plastic product. To get round the difficulty, use has been made of the influence of bulky side groups on polymerizing butadiene with a certain amount of styrene, the resulting copolymer butadiene-styrene, Buna S, has rubbery properties and makes an excellent substitute for natural rubber<sup>35</sup>. In the light of the above considerations, it is easy to understand how the incorporation of a certain amount of bhilawan shell oil or cashew-nut shell oil<sup>36</sup>, in which the phenolic molecule has a long aliphatic side chain, can reduce the brittleness of lac moulding compositions, here the incorporated long side chain compound acting as a plasticizer and giving flexibility and toughness to the rather brittle lac resin.

The foregoing general conclusions about the dependence of mechanical properties of high-polymers on the molecular texture and ultimately on the chemical nature of the monomers from which the polymeric material is produced, as revealed by the fundamental research on high-polymers carried out during the last two decades, are in general of a qualitative and in many cases of a quantitative nature. They are not only a help to synthesize new high-polymers with desired specific properties; they indicate the need for further fundamental research in which we in India can make our own contribution.

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## Cultivation of Rosha Grass (*Motia*) & Distillation of Oil therefrom

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**R**OSHA grass generally grows wild in the forest tracts of the Bombay Province in the Khandesh, Nasik and Panch Mahals districts. Possibilities of raising the grass as a cultivated crop were studied in the Punjab at Lyallpur<sup>1</sup>, and the following observations were reported :

1. The grass can be grown successfully as a cultivated crop ;

2. it requires little attention except for burning the clumps when dry ;

3. the yield of oil was 15-20 lb. per acre, the oil content of the grass being about 0.5 per cent ; and

4. it was not necessary to sow the seeds every year.

It was thought desirable to undertake similar experiments in Bombay Province and study the suitability of the soil, yield and quality of the oil. The cultivation of rosha grass was, therefore, undertaken in the year 1942-43 in the experimental plots in the

Empress Gardens, Poona, in co-operation with, and under the supervision of, the Silviculturist of the Forest Department. The investigation was confined to *motia* variety of rosha grass as *motia* oil is superior in quality.

**Cultivation**—Initially a plot of land measuring about 1,295 sq. ft. was put under cultivation. Seeds were sown and germination was satisfactory. It was, however, found necessary to weed out the plots within about 10 days after sprouting, and once again later in the season. After the grass grew to a height of about 3', the growth of weeds was not a serious menace.

After the preliminary experimental cultivation, 5 plots were cultivated under different conditions (as detailed below) and the following observations were made during the period October 1944 and October 1947:

- (i) Weight of grass from each plot;
- (ii) percentage of blossoms and stalks in the grass and their respective oil contents (on wet basis); and
- (iii) geraniol content of the oil.

**Distillation Still**—An improved type of still (50 lb. capacity), fitted with a false bottom and heated in such a way that the flames did not reach the part of the still containing the grass, was used.

**Analysis of the Oil**—The geraniol content of the oil was ascertained by acetylating the oil and determining the ester values of the oil and the acetylated product<sup>2</sup>.

**Plot "A"**—This was the original plot in which seeds of *motia* variety were sown in 1942, the clumps being 6' apart. The blossoms obtained in 1942 and 1943 were allowed to dry and seeds collected for subsequent sowings. The average yield of grass for October seasons was about 4,000 lb. per acre, and the yield of oil was 11-17 lb. per acre. The oil content of grass slowly increased from 0.32 to 0.48 per cent during the first three years after which it decreased to 0.40 per cent.

It was observed that the grass grew vigorously in the second season and blossomed by the beginning of the following January, provided there were a few good showers after the first cutting in October. The yield of grass in January was almost half of that in October, while the oil content was 0.40-0.50 per cent.

**Plot "B"** (Area, 2,750 sq. ft.; seeds sown in 1943; clumps 3' apart)—Germination during the first year was not satisfactory,

and data were collected only from October 1944. The average yield of grass per acre for the October seasons was 11,000 lb. and the yield of oil per acre was nearly 40 lb. The oil content of grass increased from 0.30 to 0.40 per cent during the three years and remained almost the same in the fourth year. During the summer of 1945, 10 out of the 32 rows from the plot were burnt, and it was observed during the following season that the yield of grass on the burnt part was 18,920 lb. per acre as compared to 11,280 in the unburnt part, the corresponding yield of oil per acre being 65.5 lb. as against 38.6 lb. in the unburnt plot. Repeated burning at the end of the second and the third seasons did not, however, yield similar results.

**Plot "C"** (Area, 3,700 sq. ft.; cultivation through transplanting rhizomes from plot "A"; clumps 3' apart)—The yield of grass was 12,780 lb. per acre in October 1944, after which it dropped down to 4,162 lb. per acre in October 1946. The stumps were burnt in the summer of 1947, which increased the yield of grass to 9,656 lb. per acre. The oil content of the grass varied between 0.22-0.37 per cent.

**Plot "D"**: Irrigated plot (Area, 1,512 sq. ft.; crop raised from root stock)—During the first year (1944) the yield was 17,860 lb. per acre as against 8,600 lb./acre in plot "B" and 12,780 lb. in plot "C"; the corresponding yield of oil per acre in these three plots was: "B"—26.6; "C"—28.5; and "D"—38.1 lb. The effect was not marked in the subsequent years due to water-logging, and the crops were damaged.

**Plot "E"**: Effect of fertilizers (Area, 1,512 sq. ft.; crop raised from root stock; dose of fertilizer, 40 lb./acre)—From the results of the 1944 season it appeared that although the yield of grass was 18,000 lb./acre, as in the case of plot "D", the oil content was higher, being 0.35 per cent as against 0.21 per cent in plot "D". The yield of oil per acre was 62.8 lb. Unfortunately, the cultivation in this plot was affected in the subsequent seasons by water-logging.

From the data collected it was observed that the blossoms formed 20 per cent of the grass, and the oil content of the blossoms was 0.76 per cent. The weight of stalks was about 4 times the weight of the blossoms, and the quantities of oil obtainable from the blossoms and stalks separately were about the same.

The geraniol content of the oil from the various plots was 95-97 per cent; the ester values of the oil from blossoms varied between 42-54, while those from the stalks varied between 32-37.

### Summary

1. It is possible to raise two crops of rosha grass during a year, viz. the first being harvested in October and the second in the following January, provided there are sufficient rains in the autumn after the first cutting.

2. The yield of grass in January is about half of that in October.

3. The oil content of grass harvested in January is higher than that in October.

4. Irrigation and manuring increase the yield and oil content of the grass.

5. 30 lb./acre is the normal yield of oil under the soil and climatic conditions prevailing at Poona.

6. It is possible to increase the yield by about 50 per cent by such cultural operations as burning of clumps, irrigation and manuring.

7. The oil content of blossoms is considerably higher than that of stalks.

### Acknowledgement

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## A Note on the Tin Deposits of India

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**T**IN deposits of economic importance are few and sparsely distributed in India. Tinstone is rarely found in peninsular India, but in the north, mostly in Bihar and Bombay, the occurrence of cassiterite has been reported from a number of localities.

Deposits from the following localities have been investigated from time to time but none of them have been exploited to any great extent. So far no systematic prospecting has been undertaken to discover new deposits or to properly assess the extent of the known deposits in the country.

### Bihar

(1) *Nurgo or Nurunga (Hazaribagh district)* — McClelland<sup>1</sup> reports the occurrence of tinstone in this area. Mining operations were started in 1867 but were later abandoned as the area proved unproductive.

Mallet<sup>2</sup> has given a brief account of this deposit. The ore occurs in 3 or 4 lenticular

beds or nests in the gneiss, which are seldom more than a foot or two across. In one or two instances they were as much as 13 feet across and extended for 20 yards in a direction parallel to the bedding of the gneiss. The ore consists of gneiss with thickly disseminated crystals and grains of tinstone. At a depth of 20 yards the nests appear to be thinning out, the rock becoming harder. The mine was abandoned as extraction was rendered difficult on account of water in the mine. Brown<sup>3</sup> described this deposit as a thin layer of cassiterite-bearing granulite in a much thicker band of microcline granulite. Ball<sup>4</sup> observed that the ore occurred disseminated in gneiss and the weathered-out tinstone gravel occupied a zone 100 yards wide extending to an unknown distance with the strike of the strata. Oates<sup>5</sup> has described his preliminary prospecting experiments in this area: "A shaft was sunk to a depth of 614'. The ore body was cut out

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and no ore was encountered below 568'. The tin content in the ore was 1.87 per cent and estimated yield of the metal was 268 lb. per ton of the ore, but in reality it was only about half of this as the ore was contaminated with much gangue and iron."

(2) *Shimratari (West of Pihra, Hazaribagh district)* — Cassiterite was found by Mallet<sup>2</sup> in this area associated with lenticles of granite enclosed in mica schist. This occurrence suggests that tinstone is associated with granite in this area.

(3) *Pihra (Hazaribagh district)* — Cassiterite occurs in a dyke of lepidolite granite a little to the south-west of this locality<sup>4</sup>.

(4) *Chappatand (Hazaribagh district)* — In this locality tinstone is found in a granulite<sup>6</sup>.

All the above localities lie in the mica belt around Gawan.

(5) *Silli (Ranchi district)* — A few crystalline lumps of cassiterite have been found to occur in a pegmatite intrusive in granulite in this area<sup>7</sup>.

(6) *Chhakkarbendah (Gaya district)* — Tinstone has been found to occur a few miles to the north of the village Chhakkarbendah<sup>7</sup>. Wolfram is associated with it. The rocks are mostly Archaean schists and gneisses intruded by pegmatite, aplite, etc. The vein containing tin has been traced for over a mile, the width varying from a few inches to a few feet.

Exploratory mining operations were commenced during the year 1940 and analytical work on the samples from this locality showed the cassiterite percentage in the rock to be 1.5.

(7) *Dhanras Pahar (Gaya district)* — Ray and Nag<sup>8</sup>, while studying the geology and the mineral resources of the Deo-Raj Estate, found tinstone in this locality.

(8) *Kalikapur (Singhbhum district)* — The presence of cassiterite has been reported recently in this region.

## Bombay

(9) *Hosainpura (Palanpur State)* — Cassiterite was found to occur with gadolinite in a tourmaline pegmatite<sup>9</sup>.

(10) *Dambal Hills (Dharwar district)* — The gold-bearing sands in the area are reported to contain a mixture of tin oxide and metallic copper<sup>10</sup>.

Of these deposits, those occurring in the Hazaribagh district have been worked to a certain extent. Little is known about the mining possibilities of other deposits.

The most promising deposit is that found in the Chhakkarbendah area.

The recent report of the occurrence of cassiterite in the neighbourhood of Kalikapur (Singhbhum district, Bihar) has encouraging features. It is interesting to note that the copper belt of Singhbhum passes close to this area and cassiterite may be associated with the copper deposits as in the case of the copper and tin deposits of Cornwall; abundant deposits of tin may be found deep down the copper veins.

The author desires to place on record his indebtedness to the U.P. Government for the award of a research scholarship to enable him carry out this survey.

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# REVIEWS

**Odors : Physiology & Control**, by Carey P. McCord & William N. Witheridge (McGraw-Hill Book Company Inc., New York), 1949, pp. x+405. Price \$6.50.

ODOURS MAY BE PLEASANT OR UNPLEASANT, attractive or disgusting. There is no common agreement on this point. The odour of garlic and onions may be more appealing to some people and very repulsive to others. Rotten cheese is liked and relished by some gourmands whereas others run away from it with a sense of nausea. The sense of smell depends upon the immediate and personal experience of "association" for the determination of pleasure or pain, attraction or repulsion by arousing past memories. "A misplaced aroma is an anathema and the lack of aroma is intolerable."

Man is said to be commonly endowed with five senses: sight, hearing, touch, taste and smell. Of these, sight is known to be caused by light waves which are vibrations in the ether, whilst hearing and touch are caused by pressure differences. Sound waves in the air impinge on the ear and bring about rapid changes in pressure on a diaphragm which vibrates and transmits the sensation of hearing to the mind and touch is caused by pressure applied to practically any part of the skin. There are definite mathematical laws as to harmony and melody in sound and colour which affect animals and ourselves to a large extent similarly. Taste and smell, however, are not induced by any known waves in the ether or in the air, but are caused by contact in the case of taste and nearness in the case of smell of certain specific substances. It is a very general experience that odours are more efficient in arousing memory than are mere colour effects or sounds. It is due to this close association with memory (conscious or unconscious) that an odour is agreeable or disagreeable. There are other factors that come into play. Taste and smell are classified as chemical senses by Moncrieff.

Continuous investigations have been made on the subject of odours in Western countries and a large volume of literature has come

into existence. That the science of odour or smell is a very complicated phenomenon is proof of the fact that an extensive vocabulary has come into daily use when anybody wants to describe the smell or odour. The following are some of the words used in literature in relation to odour: "smells may be pleasant or unpleasant, feeble, faint, strong, bland, pungent, mawkish (Scotch mawk — maggot), fruity, dank, arid, stuffy, mephitic or sulphury, aromatic, ethereal, balsamic, acid, fragrant, ambrosial, burnt, repulsive, nauseating, foetid, penetrating, distracting, alluring, seductive, soft, heavy, sharp, alliaceous (smell of garlic), rancid, spicy, flowery, resinous, foul and so on" (Moncrieff). The various nuances of the perception of smell can thus be imagined depending on the personality of each man and the mental and emotional experience or associations he has had under different circumstances. Even metaphorically, words generally connoting "the odour" have crept into every literature of the world. The use of the word "odour" to emphasize past associations comes to a grand climax in the Sanskrit expression — पूर्व जन्म वासना (the haunting odour of a past existence or previous birth).

To produce the sensation of smell in animals and more specially in man, the quantity of the substance which is responsible for the smell is infinitesimally small. The most well-known example quoted in textbooks of science is that of musk. Statements have been made that a small speck of musk continues to emanate its smell for years continuously and still not lose any weight as can be measured by the most sensitive chemical balance in existence.

A great deal of work has been done recently on the subject of "threshold concentration", i.e. the minimum concentration for any particular substance which just arouses the sensation of smell in human beings. Two or three examples may be quoted. The concentration of odori-vector in air wt./wt. per cent in the case of chlorophenol will be  $3 \times 10^{-7}$  (corresponding to 0.004 mg. in a cubic metre of air); in the case of mercaptan it is  $3 \times 10^{-9}$  (0.00004 mg. in a cm. of air);

in the case of synthetic musk it is  $4 \times 10^{-10}$  (corresponding to 0.000005 mg. in a cm. of air); it is, in the case of vanillin,  $2 \times 10^{-11}$  (0.0000002 mg. in a cm. of air). These examples should suffice (Moncrieff). The importance of these estimations lies in the fact that when we have to consider the nuisance value of disagreeable odour in the life of the community, the actual quantity of a substance that goes to create the nuisance may be exceedingly small and may still be a nuisance. Purely from a physiological point of view, such small quantities of odorous substances permeating the air may not be harmful but they still create a horrible nuisance.

From the point of view of the life of a community, an odour—that which acts on the special nerves of smell distributed in the chambers of the nose—acquires its attractive or repulsive quality only as a result of mental association with what is beneficial (suitable food, mates, friends, safety, home, the nest) or what is injurious (unsuitable food, poison, enemies, danger, strange surroundings, solitude). These are conditioned as a result of tradition or through personal experience to be beneficial or injurious and hence determine man's liking for and choice or rejection of odours and flavours. For example, the odour of sewer gas, foul accumulations of refuse, or putrescence has become painful and almost unbearable to the more cleanly classes of mankind owing to the association with it, or as a result of education, of the fear of disease and poisoning. Those members of the despised profession, but none the less the real and true public servants, viz. those that are engaged in scavenging work or those that work in the so-called offensive trades and industries, get used to mal-odorous surroundings, and are known to eat their meals with gusto and relish even when on duty at mid-day intervals. It is but right and merciful that God grants some indescribable immunity to these noble public servants!

Several distinct and repulsive smells liable to occur on the human body are generally due to want of cleanliness. The fatty secretions of the skin are often decomposed by bacteria even before complete extrusion from the glands in which they are formed, as for example, perspiration. Through perspiration the skin gets rid of water, inorganic salts, traces of urea, certain odorous principles of the food eaten

(like the smell of garlic) and others. They will undergo bacterial and chemical changes—and contribute to what is popularly termed as "body odour". This body odour differs from person to person according to habits and according to different races and communities. But in the young and healthy animals in natural conditions (including man) there is some check on the putrefying activities of the omni-present bacteria, or decomposition due to chemical changes. Generally speaking, the skin of a healthy, youthful adolescent has a pleasant odour and the breath is naturally sweet smelling. These factors have an important bearing on the personal hygiene of human beings congregated in large communities and also the proper washing and treatment of the clothes they wear.

The foregoing general remarks on "odours" will give a general background of the various aspects of the subjects dealt within the book under review. In the introduction to the book, they state as follows: "Granting endless opportunities for disagreement, for the objectives of this book an offensive odour is one that, from its own properties, chemical and physical, is disturbing to the majority of healthy persons exposed to its olfactory action. . . . Without, on the one hand, condoning all the disturbances attributed to offensive odours or, on the other, rejecting all significance of offensive odours, this has been devised to make available, in objective fashion, personal and accumulated experience related to odours in general but chiefly to the offensive ones, their control, and their import in the human communality."

In other words, the aesthetic and beneficial contribution of odours (such as perfumes and cosmetics) as social embellishments to make one's self more pleasant or attractive in society do not form a serious part of this book. Nor is the significant part that odour plays in biological functions such as mating and the preservation of species, struggle for existence, etc., given any prominence in this book. In a community, offensive odours from solid, liquid and gaseous wastes are always present and the abatement, masking or elimination of such odours for the general well-being of the community from the public health point of view happens to be the main objective. From this point of view this book may truly be termed as a "community service book". Every practising doctor,

every public health officer, anywhere in the world, will derive benefit by going through the pages of this book.

The book contains 23 chapters and an extensive bibliography. Starting with the anatomy and the physiology of the nose, all the subjects are historically dealt with in a most logical sequence with ample documentation. The next 3 chapters deal with the chemical constitution of odours, their classification, their detection and measurement. A good amount of work has already been done on the theory of odours. Starting from 1870 till 1947, there have been 23 theories on the subject and no agreement seems to have been finally arrived at. An odour may be due to the volatility of its constituent or constituents, solubility, chemical reactivity and to molecular vibrations. According to Dyson, an odorous substance must have appreciable vapour pressure so that the odorous molecules may make contact with the olfactory region. It must be soluble in the liquid matter of the olfactory region. It must have intra-molecular vibrations of such a period that it has Raman shifts between  $1,400\text{\AA}$  and  $3,500\text{\AA}$ . The tendency is to attribute the causation of an odour to intra-molecular vibrations while giving a minor but, none the less, a contributory place to other chemical and physical properties of the odour substance. The existing knowledge on the subject is ably reviewed by the authors in different chapters. After this, the place, functions and the influence of odours in relation to the well-being of the community and their control are dealt with according to a well thought-out plan, viz. the authors start with human beings, their personal hygiene in health and disease from the point of body odours; next in importance comes the home of a citizen and the subject of household odours are dealt with; next in order, the primary needs of human beings, viz. food and water, are dealt with from the odour point of view (including municipal drinking water supply and sanitary services). Human beings congregate in large offices for their daily work and also in industrial establishments that carry on processes which come under the category of offensive occupations — these are dealt with next. The question of proper ventilation and air-conditioning both in an ordinary household and also in large factories and offices are dealt with, with hints to architects and engineers in the

designing of these buildings. Whether it be in one's own household or in a factory or the office, if the offensive odours should be controlled, abated, masked or eliminated, certain steps have to be taken to achieve the object in view and the authors have a number of suggestions which have proved effective in practice. Their own personal experience and achievements are recorded with a certain amount of modesty. For example, the treatment of sewage in a city in the most innocuous manner and the treatment of the city drinking water supply by coagulants, chlorine or activated charcoal, etc., are dealt with comprehensively.

Finally, the legal aspect of the odour nuisance has been dealt with according to the American Law. The misgivings and misapprehensions which the ordinary man in the street has regarding the nature, character and influence of offensive odours have been carefully analysed. This is the first time that the legal aspects of a subject which has been the basis of scientific investigations in the theoretical and practical fields have been dealt with in a scientific text-book.

The authors deserve our thanks and our meed of congratulations. They have done the job systematically and thoroughly. It must have involved a very close and exhaustive study of the subject over a number of years and it is a boon to the community that such an excellent treatise should be made available to those who are interested in this subject. This would include doctors, public health officers and legislators and administrators. None the less, even an ordinary householder has a great deal to learn by reading some of the chapters of the book dealing with household and body odours.

One gets the impression on going through the book that special emphasis has been laid on human body odours, specially human sweat or perspiration. This may be looked upon as an indirect indication of the specially developed fastidiousness in matters of personal hygiene noticeable in American society. Personal cleanliness, subjectively, may bring about an agreeable sense of well-being and one may enjoy the luxury of the self-complacent thought that cleanliness is next to Godliness! But, objectively, from the detailed treatment of the subject in the book, the authors suggest that personal cleanliness is a social necessity. "Who dares disagree with them?"



Just a word about the bibliography. The text of the book covers 272 pages and the bibliography covers another 125 pages. As the authors claim, this may be the most complete bibliography in the world in English language. Even granting this, this chronicling and recording of anything and everything of the subject has, in my opinion, been overdone. There is no special point in trying to be thorough and too complete. In books of this nature, one need only mention important references leaving out other minor scientific communications, which may find a place in scientific journals when one special or distinct aspect forms the subject of a specialized investigation. There is also no special merit in making the bibliography too long and too exhaustive in a text-book of this nature. In this case the extra 125 pages must have added considerably to the cost of publication and the consequent increase in the price.

Finally, I would like to mention that this book will form a companion volume to another excellent earlier British publication entitled *Chemical Senses* by R. W. Moncrieff, published by *Leonard Hill Ltd.*, London, 1944.

I acknowledge with gratitude that I have made use of, and freely borrowed from, the literature on the subject available to me at Bangalore.

S. G. SASTRY

**Theory of Groups and Its Application to Physical Problems**, by S. Bhagavantam & T. Venkatarayudu (Andhra University, Waltair), 1948, pp. xi+234. Price Rs. 20.

THIS BOOK BY TWO AUTHORS WHO HAVE themselves made contributions towards the application of group theory to crystal physics is a valuable addition to the growing literature on the subject. The two main branches of theoretical physics wherein the theory of groups and group representations finds numerous applications are molecular, specially crystal physics, and the relativistic quantum theory of elementary particles. In the former the vital rôle is played by the rotation and crystallographic groups, and in the latter the Lorentz group, conditioned as the theory is by the requirements of Lorentz invariance. It is perhaps right to say that the book before us is mainly devoted to the former aspect of the physical applications.

In this particular field in which the interest of the authors mainly lies, the exposition of the several topics is very lucid, and is bound to be of great use to those who want a rapid introduction to the subject, but have no time to study the more systematic treatises.

The bare outlines of the theory of groups are given in the opening chapters, and the fundamental theorems, although not fully proved, are effectively presented with an eye towards applications. We note a slight lapse in exposition in the definition of isomorphism of two groups on page 24, para 3. The correct definition can be found in Van der Waarden, *Moderne Algebra*, Vol. 1 (2nd edition), page 31. A brief and clear account of some irreducible representations of the rotation and Lorentz groups is given in chapter 12. The chapters on spectroscopy include derivations of the usual selection rules, intensities of spectral lines and discussions of electron spin and band spectra. A very good account is given of the applications to Raman effect, infra-red absorption, etc.

There is no doubt that the book will be widely used and appreciated by physicists who have occasion to work in these fields. The authors are to be congratulated for the production, and the *Andhra University* for the sponsoring of such a scholarly publication in the field of mathematical physics.

B. S. MADHAVA RAO

**Fream's Elements of Agriculture**, revised and edited by D. R. Robinson; prepared under the authority of the Royal Agricultural Society of England (John Murray, London), 1949, 13th ed., pp. xi+723; 110 figures and 58 halftone plates. Price 21s. net.

IN A RECENT SURVEY CARRIED OUT IN Cambridge, it was found that nearly 50 per cent of the scientific text-books recommended to the students were not available for purchase, and that nearly 33 per cent of the books so recommended had to be of American origin, as against 10 per cent before the war. This illustrates briefly, how serious is the shortage of suitable text-books in Britain, and how eagerly this new and completely revised edition of this well-known and classical text-book will be welcomed, particularly by students of British agriculture.

Fream's *Elements of Agriculture* was first published in 1892, and for the last half a century and more, it has served usefully, successive generations of students and agriculturists. The very fact that it had gone through a dozen editions by 1932 and had seen a number of reprints of each edition in between, indicates its high popularity. The last edition was edited by Sir Rowland Biffen and his colleagues of the *School of Agriculture*, Cambridge. That edition was reported to have been completely revised and entirely rewritten in the light of the great changes that had taken place in the agricultural science and in the outlook of agriculturists. The causes which justified the revision in 1932 are even more valid with the present edition, which appears after a lapse of nearly two decades during which revolutionary changes have taken place in British agriculture consequent upon the demands of World War II. Not only have high-powered machines replaced horse-drawn elements in various fields of farming, but systems of farming have been altered with a view to meet the greater demands for food crops. Bogs, fens and moors have been ploughed up and permanent pastures converted into arable lands. Ley farming, once confined to Scotland and the moist parts of England, has spread to other parts of the country. The advances in British agriculture have been so extensive and rapid, that a new edition incorporating advances in all branches of crop and animal husbandry had already become overdue.

The *Royal Agricultural Society of England* has to be congratulated for bringing out this new edition suited to the needs of post-war agriculture. The book is edited by Dr. Robinson with the assistance of a large team of well-known authorities.

The present edition follows the plan of the previous edition, but nearly all the chapters have been completely revised, rearranged and many parts rewritten in order to bring the matter up to date. No effort has been spared to bring out a readable and authoritative work suitable for both students of agriculture and farmers who wish to gain an all-round knowledge of agricultural science.

The subject-matter of the book is divided amongst 24 chapters and 5 appendices, covering in all 714 pages. The first 8 chapters deal with general agriculture. The next 8 chapters are devoted to crop husbandry

and the last 8 chapters to animal husbandry. Following the text are 5 appendices summarizing a great deal of useful information. The first one gives a list of books for further reading. It is commendable that most of the books cited are the latest available in their respective fields. Appendix 2 summarizes sowing and yield data of crops and in a way replaces the deletion of a separate chapter devoted to this topic in the previous edition. Appendix 3 deals with *Seeds Act* of 1920 and appendix 4 with the control of weeds by chemicals. The last appendix summarizes the composition and nutritive value of feeding stuffs. A comprehensive index completes the volume.

The present edition possesses a number of useful features which are an improvement over the previous one. Amongst these, the most important are the inclusion of a new chapter on farm buildings and permanent equipment and the stress laid on the use of mechanized farming, particularly tractor farming. A rearrangement of the chapters has been made, particularly on crops, resulting in a more rational treatment of the subject. The chapter on insects which was at the end of the book in the previous edition has been shifted to a more logical position following the diseases of crops. The chapter itself has been considerably enlarged and copiously illustrated to include all the pests of crops and stock. Among other features may be mentioned the inclusion of such topics like the rôle of trace elements in plant and animal nutrition, use of composts, weed control by chemical methods and a greater emphasis on cytological details in plant breeding.

It is obviously difficult for a single reviewer to adjudge fairly a book which covers such a wide range of subjects. All the sections appear to have been brought up to date and much of the information that is new fully incorporated. While it is difficult in a book of this type to satisfy all the readers, it appears to the reviewer that the book could have been slightly enlarged, without increasing the bulk considerably and a short chapter included on farm management, in view of the changes brought about by extensive mechanization. A section on bee-keeping would have been useful in view of the importance attached to it as a subsidiary agricultural occupation. The chapter on animal production and dairy products included in the previous edition could have been usefully retained,

at least in an abridged form. The list of books intended for further reading could have been slightly enlarged and given under the appropriate chapters, as was done in the previous edition, rather than cited in an appendix. The index itself could have been made more exhaustive in order to include all the important topics and plant and animal names cited in the text, particularly in view of the absence of a detailed list of contents as in the previous edition.

In spite of these omissions, the book is comprehensive and provides an excellent introduction to students of agriculture. Although it is addressed to English students and farmers, the lucid exposition of the fundamental principles gives the book a much wider field of utility. The book is well printed and profusely illustrated. The revised edition maintains in all respects the high standard of its predecessors and is sure to enjoy a further span of fruitful life in agricultural education.

K. R. RAMANATHAN

**Progress in the Theory of the Physical Properties of Glass**, by J. M. Stevels (Elsevier Publishing Company Inc., Amsterdam), 1948, pp. xi+104. Price 10s.

THIS PUBLICATION IS ONE OF A SERIES OF monographs on the progress of research in Holland during the war, and is based on the results of investigations planned and carried out in the *Philips Research Laboratories*. The work reported upon relates to some important physical properties of glasses, particularly those which are understood to be correlated with the structure of glass at room temperature, such as density, dielectric losses, electrical conductivity and molecular refraction.

The first chapter of the book, viz. general introduction, gives an excellent résumé of the views so far propounded or currently held and our state of knowledge concerning structure in the vitreous state. It is largely based, therefore, on the ideas set forth by Zachariasen, and which found confirmation in the experimental work of Warren and his coworkers. As stated by Zachariasen, the atomic or molecular arrangement in the vitreous state is characterized by an extended network which lacks symmetry and periodicity. This is in strong contrast with the regular arrangement in the crystalline

state and leads to the characteristic behaviour of glasses as super-cooled liquids in which, although the crystalline state is thermodynamically the more stable one, the mobility of the atoms has become so small as to disallow the possibility of regular packing taking place. Zachariasen concluded that the free energy of the solid glass cannot exceed that of the crystalline phase by a large amount and his conclusions have proved to be an excellent guide to subsequent workers in their investigations on this subject.

For calculating the densities of glasses from chemical compositions, a simple additive formula has been in vogue. On the basis of the work reported in the book, a new method, more closely connected with an insight into the structure of glass, has now been developed. In chapter II, dealing with density, the validity of the new method has been considered from the viewpoint of structure in the vitreous state, and its applicability to the cases of one-component and multi-component glass systems has been illustrated, and discussed in considerable detail. On the basis of conclusions formed from considerations of density, a more precise understanding of the phenomenon of electrical conductivity in glasses as a function of temperature and composition is discussed in chapter III, and an explanation is advanced for the interesting results obtained by Gehloff and Thomas that although electrical conductivity in glasses is believed to be entirely due to the movement of  $\text{Na}^+$  ions in silicate glasses with a constant total weight percentage of  $\text{Na}_2\text{O} + \text{K}_2\text{O}$ , such conductivity does not increase continuously on replacing  $\text{K}_2\text{O}$  by  $\text{Na}_2\text{O}$ , but decreases slowly to a minimum value and then increases rapidly.

Dielectric losses of glass form the subject-matter of chapter IV. This is a subject on which there is very scanty literature and hardly any systematic work has been carried out before except that by Strutt, who gave an empirical relationship between the power factor and temperature involving constants whose values were dependent upon the composition of glass and the frequency. A theory governing such dielectric losses has been developed by the *Philips'* workers on the basis of the structure of glass, and it has been surveyed in the light of experimental results. The last chapter gives a similar treatment of molecular refraction for which it had been found

by earlier investigators that experimental values often differ from the values calculated by adding the atomic refractions which are independent of the composition.

Altogether, this monograph is a very valuable contribution to the literature on glass in English language. Dealing as it largely does with a terse and speculative subject, viz. structure in the vitreous state, and an understanding of the physical properties in terms of structure and chemical composition, it may find but a limited circle of readers.

The numerous symbols and abbreviations used and what they stand for are rather difficult to remember as one progresses through the book but perhaps this was inevitable in a research publication of this nature. It will find invariably a place in the library of every research organization dealing with glass technology.

The monograph has been well printed and neatly got up in a handy size.

Y. P. V.

**Properties of Soft Solders and Soldered Joints (Research Monograph No. 5),**

by J. McKeown (British Non-Ferrous Metals Research Association, London), 1948, pp. 140 + 56 illustrations + 49 tables. Price 17s. 6d.

THE BRITISH NON-FERROUS METALS RESEARCH Association has, from its first days, taken an interest in the problem of solders and soldering. The results of its first investigation appeared in 1932 in the Association's Research Monograph No. 1, *Tin Solders* by S. J. Nightingale, a second edition of which, revised by O. F. Hudson, appeared in 1942. Meanwhile, the tin shortage following the beginning of the war in 1939 stimulated the B.N.F.M.R.A. (which was continuously consulted on methods of economizing in the use of this metal) to researches on solders of lower tin content.

Information was lacking on the mechanical properties of the tin-economy solders

and joints made by them. To fill this gap, a wide variety of tests were carried out over a series of temperatures including tests on the creep properties of joints made with a range of solders. In addition, comparison was made (by soldering, area-of-spread and capillary penetration tests) of high-tin and tin-economy solders. The hot tearing of solders was examined in order to define suitable compositions for the avoidance of this trouble; and investigation was made of the soldering of aircraft oil-coolers by lead-rich solders.

This experimental work and the conclusions drawn from it are described in the present volume by Dr. John McKeown who led the team concerned with this war-time research.

The monograph is divided into 6 chapters.

The first chapter deals with the properties required in solders and solder joints. Chapter II is devoted to composition of solders, under which head description of the former British standards, tin economy solders and solders investigated during the war years are given. Various soldering power tests are dealt with in the third chapter. This again is sub-divided into 3 sections: (i) bit-soldering tests; (ii) area of spread tests; and (iii) capillary penetration tests. Chapter IV deals in 5 sections mechanical properties of solders and joints, effect of addition of tin to lead-silver solders, life and creep tests on joints and fatigue tests on lap joints. The last two chapters deal with hot tearing in solders and the soldering of aircraft with lead-rich solders.

The investigation described in the present publication and the conclusions reached are of permanent value as a substantial addition to knowledge of the soldering process and the properties of behaviour of solder joints. The volume should prove valuable to all users of solders.

The get-up and printing of the book are of a high order.

A. K.

# Fluorine Chemistry

M. M. SINGH

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**F**LUORINE and its compounds have unique properties due to the fact that fluorine occupies the extreme upper right-hand corner of the Periodic Table and is much more electro-negative than its neighbours. The compounds of fluorine—both organic and inorganic—due to their extreme properties not only test and extend our theories of chemical combination, structure and reaction, but provide a large number of new substances of varied utility. Though prepared first in 1886 by Moissan<sup>1</sup>, it remained, for many years, a mere chemical curiosity. It is only after the first quarter of the present century that the chemistry of fluorine attracted serious study. It received a great impetus during the last war in view of its bearing on the development of atomic energy. Fluorine in the elemental form was first put up for sale in the American market<sup>2</sup> in 1946, and it is likely to become available in England this year.

## Inorganic Compounds

A large variety of inorganic fluorine compounds is known. Some of them have established large-scale uses. A few examples will suffice: fluorite in metallurgy, particularly of iron; cryolite in the metallurgy of aluminium; sodium fluoride and barium fluo-silicate as insecticides. Liquid hydrogen fluoride provides a medium for the liquid phase oxidation of organic compounds with molecular oxygen as the oxidizing agent<sup>3</sup> at temperatures below 200°C. There are possibilities of numerous applications for other inorganic fluorine compounds based on the tendency of fluorides to form double and complex salts. Many crystalline compounds are formed with hydrogen fluoride, such, for example, as the hydrofluorates of sodium fluoride and potassium fluoride. Many complex ions in which fluorine is involved are known. The iron-fluorine complex is the basis for the use of potassium bifluoride in removing rust spots from clothing and similar materials. Compounds of fluorine with other electro-negative elements are of great interest. Sulphur hexafluoride is a surprisingly unreactive compound, and is, therefore, useful in high voltage work<sup>4</sup>. The fluorides of bromine and chlorine, on the other hand, are highly reactive. The latter ( $\text{ClF}_3$ ) was made by the Germans in relatively small quantities for military purposes (incendiary) during the last war.

## Organic Compounds

In the field of organic chemistry, many more fluorine-containing compounds are theoretically possible than are known. At present that number is not as imposing as that of organic chlorine compounds. One reason is that the usual methods of synthesis are not always applicable for the preparation of organic fluorine compounds. A significant development in technique is the replacement of chlorine by fluorine by using silver and mercury fluorides. The Swarts reaction and the

modified Sandmeyer (sometimes called Schiemann) reactions have proved satisfactory for the synthesis of many fluorine compounds. Organic fluorides vary in activity from extreme inertness to high reactivity. In general, the introduction of fluorine imparts inertness and stability.

## Commercial Applications

The commercial applications of fluorine compounds are many. The freons, especially Freon-12 ( $\text{CCl}_2\text{F}_2$ ), are important in refrigeration and air-conditioning. During the war, Freon-12 was employed in aerosol bombs<sup>5</sup> for exterminating malarial mosquitoes in ships and barracks. In India, where vast regions are malaria-ridden, Freon should prove a real boon. Hydrogen fluoride is extensively employed by the petroleum industry in the synthesis of high-octane gasoline.

Many inorganic fluorides, notably fluorspar, boron trifluoride, cryolite and sodium fluoride have been commercially important for years. Sulphur hexa-fluoride is being used as an effective insulator in high voltage electrical and X-ray fields. Sodium fluoroacetate is a valuable rodenticide, and fluorine-containing analogues of D.D.T. have found use as insecticides.

Fluorocarbons find a wide application in industry on account of their non-flammability and extreme stability to heat and chemical reagents<sup>6</sup>. The unique solvent properties of this class of substances should make them particularly valuable in the field of solvent extraction. The fluorocarbon plastic "Teflon" is a product of considerable interest. Its use in the generation and handling of fluorine is of special interest. On the basis of information now available, it may be stated without exaggeration that in the study of fluorocarbons we are confronted with a vast new field of "inorganic-organic" chemistry which is as immense as organic chemistry.

Future developments in fluorine compounds may be expected to include: dyes, plastics, pharmaceuticals, lubricants, tanning agents, metal fluxes, fumigants, insecticides, fungicides, germicides, rodenticides, fire-extinguishers, fire-proofing compounds, solvents and heat-transfer media. All present indications point to the conclusion that fluorine chemistry is destined to become important, both from the point of view of its theoretical importance and its commercial applications.

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# Defence Research in Australia

**T**HE work of the *Defence Research Laboratories (D.R.L.)*, Department of Supply and Development, Australia, during the year 1947-48, continued its work along the lines described in the previous report. An important aspect of the research carried out by the *D.R.L.* (other than the confidential work carried out for the Armed Forces) is the prominence given to fundamental research. Other equally significant features are the amount of research carried out by the several defence research laboratories for private industry and the attention paid to long-term investigations.

The distribution of work as originating from the different groups of clients or initiated with the *D.R.L.* as long-term investigations was as follows:

Department of Supply and Development	%
and Defence Departments ...	40
Other Government departments and public	
Instrumentalities ...	23
Private industry ...	16
Long-term investigations ...	21

The *D.R.L.*, maintains close collaboration with the *Council of Scientific & Industrial Research* and with the New Zealand scientific establishments.

Fees for laboratory work are charged to private firms but not to governmental authorities. They are on a nominal scale and are not designed to make a recovery of overheads, the unrecovered portion of costs representing the government contribution to the development of industry.

Electrical work had hitherto been done by the Physics Section of the *D.R.L.* but the need for a new section for electricity had become urgent, as the facilities already obtaining at the *D.R.L.* for testing, consultative, investigational and research work in electricity and electronics are inadequate for the future needs. Sanction has been obtained for establishing a new Section for Electricity.

The major activities and developments of the various sections and inter-state branches are:

**General Chemistry Section**—Specification testing and advisory work for the Services have continued to be a substantial part of the Sections' activities, i.e. identification and checking of reserve and surplus stock materials such as lubricants, corrosion preventives and other general stores.

**Chemical Defence Section**—An increasing number of requests from Government departments and industry for advice and assistance in connection with the sampling and estimation of vapours and air-borne dusts have demonstrated that the Section meets a real want in this field.

Arising from a *F.I.A.T.* report describing a German process used for the preferential recovery of ethylene from coke-oven gas, the possibility of the utilization of charcoal for this purpose has been investigated. A laboratory plant based on the continuous principle was erected and a concentration of ethylene from 4 per cent to 20 per cent has been obtained.

**Explosives & Ammunition Section**—The work of the Section during the year under review has been concerned mainly with the design,

manufacture, and functioning of ammunition, and the manufacture and storage of explosives required by the Defence Services.

**Metallurgy "A" Section**—A major proportion of the activity of the Section has been given to investigations relating to metal processing and the behaviour of metal in service.

**Metallurgy "B" Section**—Three-fourths of the routine and *ad hoc* investigations carried out by the Section has been for the Government departments and the rest for private industry. Outside activities which engage the attention of the Section include work for various Committees of the *Standards Association of Australia* and the *National Association of Testing Authorities*.

An important function of this section is the training of workers in industrial radiography for other establishments and industry.

**Engineering Section**—Both governmental bodies and private industry use the facilities available in this Section. Much of the work carried out is in connection with day-to-day problems, but long-term investigations have been an important part of the Section's activities. Fatigue characteristics of alloy steels, testing and calibration service and fabrication of testing instruments are some of the typical examples.

**Metrolgy Section**—The Section carried out a large amount of general examination and *ad hoc* investigational work on behalf of Government departments and private industry in connection with gauging and measuring methods, inspection of equipment, specifications, drawings, etc.

**Physical Section**—A study is being made of the modern techniques of interference microscopy and phase-contrast microscopy. Special-purpose monochromat objectives have been designed. A survey was made of possible formulae expressing the refractive index of optical glasses as a function of wavelength. Decade counting units have been built which are reliable in operation at frequencies up to 1 megacycle per sec. A magnetostriction oscillator, consisting of a nickel tube vibrating longitudinally at about 30,000 cycles per sec., has been built for the purpose of disintegrating bacteria.

**Workshops**—During the year there has been an increase in the proportion of work of a specialized nature carried out by the experimental glass-working, fine instruments, etc., as follows:

**Glass-working**—Construction and mounting of glass membranes, production of components for microscope lenses, experimental haemocytometer, etc.

**Fine Instruments**—Construction of platinum resistance thermometers, experimental "Vortex Tube" apparatus for the liquefaction of gases, components for a mercury diffusion pump, experimental sonic disintegrator for bacteria, etc.

**Mechanical**—Equipment for optical projection, X-ray diffraction equipment, mechanical components for microscopes, pantograph hob-measuring instrument, etc.

**Technical Information Service**—This Section works in close collaboration with the other laboratory sections. The number of enquiries received during the year has shown a marked increase.

# NOTES & NEWS

## Neomycin, a New Antibiotic

THIS NEW ENTRANT INTO THE field of antibiotics, active against streptomycin-resistant bacteria including streptomycin-resistant strains of *M. tuberculosis*, has been isolated from culture media on which *Actinomyces fradii* (now *Streptomyces fradial*) was grown (*Science*, 1949, 109, 305). The bacterium, when grown on a medium containing peptone or meat extract, glucose and common salt, is found to produce the antibiotic under both stationary and submerged conditions of culture. Neomycin can be separated from the culture medium and concentrated, employing methods similar to those used in the case of streptomycin.

Neomycin is a basic compound, most active at an alkaline pH. It is soluble in water, insoluble in organic solvents, and is thermostable. It is active against numerous Gram-positive and Gram-negative bacteria, especially myco-bacteria, but not against fungi.

The outstanding properties of the new antibiotic are: (1) activity against both streptomycin-sensitive and streptomycin-resistant bacteria; (2) high potency (in some cases greater than streptomycin) against various forms of *M. tuberculosis* and myco-bacteria; (3) limited or no toxicity to animals; (4) activity against various bacteria *in vivo*, including Gram-positive and Gram-negative organisms and (5) absence of resistance against neomycin among organisms sensitive to it, or only limited development of such resistance.

When a 20-hour old agar culture of *E. coli* was suspended in water and plated out in nutrient agar containing 5  $\mu$  or 25  $\mu$  of neomycin per c.c., no colonies of *E. coli* developed out of 22 billion cells after 9 days incubation at 28°C. Similar concentrations of streptomycin would usually allow the development of a dozen or more bacterial colonies per plate.

Broth or agar culture containing sufficient neomycin to inhibit the growth of bacteria were incubated for considerable periods of time. No further bacterial

development occurred beyond a certain initial inhibiting concentration, thus pointing, on the one hand, to the stability of neomycin as contrasted with that of aureomycin and, on the other hand, to the lack of resistance developed among the sensitive bacteria, as contrasted with streptomycin.

## Pyrethrin Substitutes

THE SYNTHESIS OF NEW PYRETHRIN-like compounds has been announced from the laboratories of the Bureau of Entomology & Plant Quarantine, U.S.A. (*Chem. Age*, 1949, 60, 484). The chemical composition of the synthetic materials is stated to be identical with that of the active principle in pyrethrum. The material will not breakdown as quickly as the natural product. One of the compounds tested is reported to be 6 times more powerful in its knock-down action than the toxic principles from pyrethrum flowers.

Starting materials for the synthesis are pyruvic aldehyde and aceto-acetic ester. Basis of the first of these is propylene glycol, and the second is made from ordinary alcohol and acetic acid.

## Soil Classification in U.S.A.

THE SYSTEM OF SOIL CLASSIFICATION developed through years of research and now used throughout the United States makes it possible to apply techniques of modern agricultural science to individual farms, according to a report of the U.S. Department of Agriculture.

Because soils differ widely within any area, the success of the application of new research findings on a given farm cannot be predicted accurately unless soils at the research station and at the farm are classified in terms that permit comparison.

On the basis of field and laboratory experiments conducted during 1912 to 1935, a new concept has been formulated. The process of soil formation in different environments are so unlike that soils developed from similar rocks in different places

have widely different characteristics and behaviour.

In defining soil types, the characteristics taken into consideration are surface slope, texture, stoniness, fertility, depth, drainage, acidity, and presence of impervious layers.

More than 8,000 different soil types have been identified. In soil survey reports, the different types in a particular area, usually a single country, are classified, shown on maps, and described. A sandy loam is described as a poorly drained, acid, sandy soil, with no layers that cannot be penetrated by water and plant roots. It requires drainage to lower the water table, is deficient in plant nutrients, and needs heavy fertilization to produce high yields of most crops.

Such classification furnishes an accurate and orderly basis for assembling in usable terms the results of research and the experience of farmers. It permits prediction of crop adaptability, probable yields, and management requirements of specific areas of land. Experimental results or farmers' experience with one type of soil may have little or no prediction value for other soil types. Soil classification provides a means of showing the types of soil on any piece of property so that farmers may choose used practices that experience of research has shown to be suitable to these soil types. It has played a major role in the development of programmes for soil improvement and conservation and for the prompt and effective adjustment of agriculture to meet fluctuating economic conditions and emergencies (*USIS*).

## Deficiency in Soils

A PRACTICAL METHOD OF DETERMINING what elements essential for normal plant growth may be lacking in a soil, has been developed by the Bureau of Plant Industry of the U.S. Department of Agriculture. Research on the tobacco plant has demonstrated the possibility of detecting specific soil deficiencies through observations of plant abnormalities.

The lack of a specific element can be detected by certain typical symptoms, especially as they first appear on the plant. The symptoms produced by deficiencies fall into two groups: (1) those localized on the older or lower leaves, or more or less general on the plants, are due to lack of



nitrogen, phosphorus, magnesium, potassium and zinc—elements that are readily mobile; (2) those localized on the upper or bud leaves are due to lack of calcium, boron, copper, manganese, sulphur and iron—elements that are relatively immobile.

Local symptoms such as chlorosis of the older leaves, with or without necrotic (dead) spots, are due to potassium, zinc or magnesium deficiency. Potassium and zinc hunger are distinguished from magnesium deficiency by the development of necrotic spots at the tips and margins of chlorotic (pale) leaves. The breakdown from zinc deficiency differs from that of lack of potassium by progressing more rapidly and not being confined so sharply to leaf tips and margins. Nitrogen deficiency is manifested by general effects with the development of a light-green colour and drying of the lower leaves. Dark-green, immature plants are characteristic of phosphorus shortage.

The younger or bud leaves may become chlorotic or wilted with or without breakdown. Boron and calcium deficiencies both result in a die-back of the terminal growth but produce different symptoms in the early stages. Calcium shortage first becomes apparent in a light-green colour, followed by a typical hooking downward, with necrosis (dead tissue) developing at leaf tips and margins; if later growth takes place, these tips and margins are missing. Boron deficiency also first appears as a light-green colour, but this is followed by more or less breakdown at the base of the bud leaves; these may later make some distorted growth but the leaves break easily and the vascular tissue is black. Copper shortage is characterized by a permanent wilting of the upper leaves; if this occurs after the flowering stage, the seed stalk is unable to stand erect.

Manganese, iron and sulphur deficiencies produce chlorotic effects on the terminal growth or young leaves without complete breakdown. In manganese deficiency, chlorosis tends to follow out the minutest branches of the vascular system, giving the leaf a checkered effect, and this is accompanied by small necrotic spots scattered over the leaf. The chlorosis due to iron and sulphur deficiencies involves no necrosis. Iron chlorosis occurs first on tissue between the veins,

but in extreme cases the entire leaf becomes white or yellow. Sulphur shortage is evidenced by loss of green on the upper leaves and by light-green veins. A deficiency of more than one element may cause a greater reduction in growth, but the most evident symptoms are commonly those typical of a shortage of the element most deficient.

These findings made it possible to prepare a key to facilitate diagnosis of deficiency effects on the tobacco plant, and have stimulated similar studies of other plants from which to establish a practical basis for compounding corrective fertilizers (*USIS*).

### Detection of Sulphur

WHEN ORGANIC COMPOUNDS containing hydrogen are heated above the melting point with elementary sulphur, hydrogen sulphide is one of the decomposition products. Its evolution is marked when a polyhydric phenol, for example hydroquinone or pyrogallol, is the reducing agent. This reaction affords a means of detecting sulphur quickly and conveniently in complex or coloured materials, provided they are not too readily combustible (*Nature*, 1949, 163, 537).

A small quantity of the substance to be tested is mixed in the dry powdered state with an equal weight of a polyhydric phenol, for example hydroquinone or pyrogallol, and the mixture heated in a bulb tube above the melting point. A freshly moistened lead acetate paper held in the issuing vapours is stained brown when sulphur is present. Under such simple conditions, it has been found that 0.0001 gm. of sulphur can be detected, but it is possible that an even higher degree of sensitivity might be attained.

Studies with a few typical sulphur compounds have shown the metallic sulphates, sulphides and sulphonates are unreactive, while sulphites are slowly reduced and yield hydrogen sulphide after a period of heating. Thio-urea yields hydrogen sulphide; presumably, any organic sulphur compound which decomposes on heating responds to the test.

The test promises to be of value in the examination of pigmented materials, dyestuffs and carbonaceous matter and, although it has obvious limitations, may prove useful in qualitative analysis as a confirmatory test for free sulphur or as an aid in the

identification of organic sulphur compounds.

The conversion of sulphur to hydrogen sulphide with polyhydric phenols appears to be quantitative and the reaction can be made the basis of a method for estimating sulphur.

### Amino Acids as Fat Stabilizers

THE COST OF STABILIZING LARD and other animal fats by means of phenolic anti-oxidants may be reduced by the use of methionine and other relatively inexpensive amino acids to synergize the activity of such costly anti-oxidants as  $\alpha$ -tocopherol, hydroquinone and nordihydroguaiaretic acid. It has been found that 0.01 per cent of an anti-oxidant together with 0.01 per cent of amino acid is as effective as 0.1 per cent of the phenolic anti-oxidant alone.

With  $\alpha$ -tocopherol as the primary phenolic anti-oxidant, the most effective synergists were found to be methionine and ascorbic acid. But with hydroquinone and nordihydroguaiaretic acid, methionine gives the maximum effect. Other amino acids, reported to be effective but not as effective as methionine, include threonine, leucine, norleucine, valine and cysteine and phenylalanine (*Chem. Eng.*, 1948, 55, 19).

### Aluminium Welding Alloy

A NEW LOW TEMPERATURE, aluminium welding alloy, Eutecrod 900X, that will not warp or distort aluminium surfaces, has recently been developed by the *Eutectic Welding Alloys Corp.*, New York (*Chem. Age*, 1949, 60, 363). The alloy will permit the welding of thin aluminium parts at temperatures below the metal's melting point with greater fluidity than is attained by aluminium brazing. The low temperature operation is said to make it particularly suited for the manufacture of all light-gauge aluminium parts, tubing, sheets, bands and shapes without risk of distortion.

The new alloy is available in  $\frac{3}{16}$ " and  $\frac{1}{32}$ " in coil form. It bonds at 900°F. and re-melts at 950°F. It has a tensile strength of 30,000 p.s.i. Corrosion resistance is stated to be comparable with aluminium and colour match is excellent.



## Amino Acid Antagonist

INTEREST OF MICROBIOLOGISTS and nutrition experts has been attracted to  $\beta$ ,2-thionylalanine since the compounds has been found to possess specific activity against amino acids. It is used in medical and biological laboratories as a specific antagonist toward phenylalanine in the metabolism of a number of organisms. This white, soluble, odourless powder is now available for stock delivery from *Arapahoe Chemicals Inc.* of Boulder, Colo. (*Chem. Eng.*, 1949, 56, 171).

## Preparation of Ammonia Solutions

A SIMPLE PROCESS HAS BEEN developed for the preparation of aqua ammonia by direct line mixing of water and anhydrous ammonia as the ammonia is unloaded from tank cars. Equipment requirements are small; the process is simple and automatic; and it makes possible attractive savings for large-scale users of aqua ammonia in that it eliminates the cost of shipping 4 tons of water with every ton of ammonia (*Chem. Eng.*, 1949, 56, 127).

Anhydrous ammonia, as it flows from tank cars under its own pressure, is mixed with an automatically controlled flow of treated water in a globe value line mixer. The proportioning of the flows of ammonia and water by means of flow instruments was considered impractical because of the difficulties in accurately measuring two-phase flow. Flow is, therefore, proportioned on the basis of the temperature rise resulting from the exothermic heat of solution. With this method of control, ammonia flows freely and uncontrolled from tank car to storage and is line mixed with a controlled flow of water; water flow is automatically adjusted to hold a fixed temperature after mixing.

The theoretical temperature rise which occurs when anhydrous liquid ammonia at 68°F. and water at 68°F. are mixed, has been calculated for several aqua ammonia concentrations from the thermal data.

Typical installations using 3" piping for liquid ammonia flow and 4" piping for water and aqua ammonia flow permit the unloading of a 10,000 gal. ammonia tank car in 6 hr.

## Effect of "Gammexane" on Paper

THE USE OF GAMMEXANE in preserving valuable documents and books in libraries has been studied from the point of view of its possible effect on the durability of paper (*Nature*, 1949, 163, 607).

No. 2 "Gammexane" smoke generators, weighing approximately 2 oz. per 1,000 cu. ft. as recommended by the manufacturers, *Imperial Chemical Industries Ltd.*, for insects infesting stored products, was employed. Samples of paper were exposed to the smoke for 72 hr. in order to allow it to settle down completely, and care was taken to prevent any scorching effect from the smoke generator. Samples exposed to "Gammexane" smoke were also submitted to accelerated ageing, which consisted in heating the samples for 72 hr. at 100°C. The tensile breaking strength and the folding endurance of the treated and control samples were measured according to the standard *TAPPI* methods.

The mere exposure of paper to "Gammexane" smoke brought about a substantial decrease in its tensile strength and folding endurance—as much as 53.7 and 71.5 per cent respectively in one instance. Papers exposed to "Gammexane" and then submitted to accelerated ageing turned slightly yellow, and there was a complete loss of folding endurance in nearly all cases. All-rag hand-made paper of an extremely durable quality used for the repair of old manuscripts had a normal folding endurance of 4,164; but on exposure to "Gammexane" smoke the retention of folding endurance was 28.5 per cent only, while on accelerated ageing, the retention of folding endurance further decreased to zero.

The data obtained conclusively prove that treatment with No. 2 "Gammexane" smoke generator is definitely injurious to paper records of all kinds and effects their durability as badly as acidic gases. As the water through which the smoke had been bubbled was found to be distinctly acidic, it is almost certain that at least a part of the embrittling effect produced on paper is due to the smoke-generating materials rather than to the "Gammexane" itself. However, the gases obtained from the smoke generator No. 2 has

deleterious effect on paper, and it is advisable to prevent it coming into direct contact with paper records and manuscripts intended for permanent preservation.

## Sunflower Oil

THE SUNFLOWER IS BEING increasingly grown in Britain as a source of vegetable oil. It needs no manuring and usually outgrows any weeds, resists drought and yet stands up to heavy rain. The yield of seed (oil content, 33 per cent; protein content, 30 to 40 per cent) is about 1 ton per acre. The oil is rich in Vitamin E.

## Plochere Colour System

THE PLOCHERE COLOUR SYSTEM is a collection of 1,248 painted cards, each 3" by 5" in size, accompanied by an explanatory book, and intended mainly for the use of decorators. It is a pigment-mixture system, and the explanatory book on the subject gives directions for each of the 1,248 colours by the mixture of one or more of nine chromatic pigments with black and white. The nine basic pigments and the special black are obtainable from the publishers of the system. The colours are classified into 26 blocks of 48 each, each set being intended to represent 1 hue, beginning with yellow and proceeding through orange, red, purple, blue and green to a greenish yellow. It is stated that these hues represent the 24 hues of the Ostwald colour circle and 2 additional hues on either side of yellow, which were felt to give an improved gradation.

Each block of 48 colours is divided into 6 series of 8. Each of these series is intended to be at a certain chrome level, the first being the strongest in colour and the sixth the least saturated. In each series the first colour has the lowest value, the remaining 7 being produced by extending this first colour with increasing amount of white. (*Can. J. Res.*, 1949, 27, 1).

## Production of Titanium

AN IMPROVED METHOD OF MAKING pure ductile titanium with possible commercial significance is being worked out by the *Bureau of Mines* at Boulder City, U.S.A. A maximum of about 100 lb. is produced daily in a pilot plant by reducing titanium chloride

with metallic magnesium. Until recently, pure titanium was considered a laboratory curiosity.

Reduction is carried out in an air-tight iron container from which all oxide has been purged by hydrogen at 500°C. Magnesium ingots are introduced through an inlet tube, and the apparatus is heated to 150°C. under reduced pressure to remove absorbed gas. After the container has been blanketed with helium, the temperature is raised to about 750°C. Liquid titanium chloride is added slowly, then more rapidly as the reaction proceeds. The heat evolved is sufficient to maintain the temperature for about two-thirds of the run. A maximum temperature of 900°C. is held for a full half hour after all titanium chloride has been added.

When cool, the charge is removed, leached with cold hydrochloric acid, dried, finely ground, leached with 10 per cent hydrochloric acid and finally washed and dried at room temperature. The titanium is pressed and sintered for 16 hr. at 1,000°C. under vacuum (*Chem. Eng.*, 1948, 55, 59).

### Fish Albumen

A WHITISH POWDER, "EWEISS", produced from stock fish and sharks has been used as a substitute for egg-white. The sensational value of eiweiss as a substitute for egg-white is shown by the fact that 1 lb. of eiweiss is the equivalent in whipped form of the whites of 250 eggs. The nutritional value of 1 lb. of fish albumen is the equivalent of the white of 125 eggs.

Fish albumen has a wide range of uses; it has a high nutritional value and can be used for all purposes for which egg albumen is used. In some respects it is stated to be superior to egg albumen, e.g. stronger whipping power, retention of this power with constant whipping, and resistance to coagulation by heat. It finds many uses in baking, confectionery, mayonnaise, custard power, ice cream, pharmaceutical products, leather industry, synthetic resins, lacquers, foam extinguishers, etc.

The yield of eiweiss is 14 per cent from fresh fish, 50 per cent from dried fish, and 75 per cent from shrimps.

Fillets of fish are treated to remove all water-soluble impurities. Fats are then removed, and the product is then processed to render it water soluble and

spray-dried to produce a fine powder, slightly cream coloured, which, when whipped up in water, produces a smooth-textured, meringue-like mixture, free from any fish odour (*Fisheries Newsletter*, 1948, 7, 23).

### Manufacture of Fatty Alcohols

FATTY ALCOHOLS ARE NOW MANUFACTURED in the U.S.A. by reducing the esters in the presence of a reducing alcohol by metallic sodium. Research by E. I. du Pont de Nemours & Co. has shown that by suitable modifications, almost quantitative yields of fatty alcohols on the amount of sodium used could be obtained and the consumption of the reducing alcohol (alcohols other than ethanol) can be lowered markedly by adding this latter alcohol as fast as it is consumed (*C.T.J.*, 1949, 124, 329).

The method is briefly as follows: the fat used is hydrogenate coconut oil, containing about 99 per cent triglycerides, 0.75 per cent unsaponifiables and 0.25 per cent free fatty acids. It is important that the free fatty acid content be kept as low as possible since it reacts with the sodium to form anhydrous soap and represents a loss of sodium. The coconut oil is passed through a two-stage vacuum dryer to reduce the moisture content to the lowest practical level (c. 0.03 per cent). The sodium used is almost pure, contaminated only by traces of calcium.

The reduction is a batch operation carried out in a mechanically agitated reactor fitted with a reflux condenser and blanketed with nitrogen. The molten sodium is dropped (2,000 lb. per batch) into the reactor which already contains 2,000 lb. toluol, the last mentioned acting as a solvent for the reaction mass and a dispersing medium for sodium. The reactor temperature is maintained at the boiling point of toluol.

The coconut oil solution (solvent: methyl amyl alcohol) is then fed to the reactor at a rate so controlled that only a slight excess of unreacted ester is present in the vessel at any time. The reaction is exothermic and the heat is removed by refluxing the toluol, the boiling toluol being reintroduced into the reactor without chilling the mixture.

The mixture in the reactor is then dropped into a mechanically

agitated quench tank containing 17,000 lb. of water where hydrolysis occurs, the quench tank being fitted with a water-cooled reflux condenser. When the whole batch from the reactor vessel has been treated, the agitation in the quench tank is stopped and the content separated into 3 layers. The top layer comprises the product alcohol and the regenerated reducing alcohol. The middle and smallest layer is a dark, soap-stabilized emulsion of the solvent and aqueous layer. The aqueous bottom layer contains the glycerol and caustic formed in the hydrolysis.

### Newsprint from Wheat Straw

THE POSSIBILITY OF MANUFACTURING newsprint from wheat straw has been proved by large-scale trials conducted by the *Chemical Paper Mfg. Co.*, Holyoke, Mass. (*Ind. Pulp & Paper*, 1949, 3, 354). In these trials the screening system was inadequate to remove shives, and equipment was not available for multi-stage bleaching. The paper had excellent formation and was much stronger than regular newsprint.

The pulping process is simple. Bales of straw are dumped into a rotary cooker without any preparation. Cooking is effected at low temperature and pressure. The cost of chemicals is estimated at \$14 per ton without recovery, but the use of a recovery system would materially reduce this figure. A 50 per cent yield of pulp on the weight of straw has been obtained.

### Glycerine Refining

A RECENT INVENTION BY THE *Nopco Chemical Co.* relates to the refining of crude glycerine produced by saponification and alkyl esterification methods (*Chem. Age*, 1949, 60, 478).

Briefly, the process consists in separating the acidic glycerine from the esterification mass, neutralizing with alkali, heating the neutralized glycerine with ammonium chloride and removing the insolubles. Neutralizing the alkali may be effected before separating the glycerine. The refined and substantially anhydrous glycerine may be further purified by distillation, co-distillation with mineral oil, or by solvent extraction, or a combination of these.

900 parts of groundnut oil were reacted with methanol in the

ratio of 15 molecules of methanol to 1 molecule of glyceride, with 1 per cent sodium hydroxide as catalyst. The mixture was refluxed for an hour, cooled, acidified with 20.2 parts of concentrated sulphuric acid and refluxed for another 4 hr. The reaction mixture separates into layers and the lower glycerine layer was drawn off; 36 parts of barium oxide were added to the glycerine layer and the mixture heated for an hour on the steam bath. It was then cooled, 29.5 parts of ammonium chloride added and further heated for an hour. Most of the methanol associated with the glycerine was then distilled off and the reaction mass filtered. The crude glycerine obtained was distilled under reduced pressure, yielding an odourless, water-white, anhydrous glycerine of pH 7 in 83.7 per cent yield.

In the second method, the crude anhydrous glycerine was first neutralized with calcium carbonate, either before or after the removal of glycerine from the mass, and the insoluble material removed by filtration. The glycerine may be heated for a short time at about 60°C. for 30 min. on the steam bath to aid filtration. Barium chloride is added along with more alcohol to increase fluidity. This eases handling and prevents loss of glycerine. After these additions, the mass is again heated at 60° to 120°C. for 30 to 60 min. at the reflux temperature of alcohol. Insolubles are again removed by filtration and any residual alcohol distilled off.

### Improved Type of Plough

THE Indian Agricultural Research Institute has evolved a new plough which, with a single pair of bullocks, is capable of doing twice the work done by the existing standard plough.

The new plough is simple in construction and consists of two standard *desi* ploughs, suitably coupled by an iron frame-work and pulled by a single central beam. The ploughs are so spaced that identical furrows are cut, and they carry out in one operation the work which would be performed in two operations by the standard plough. The plough is comparatively light, its weight being only about 50 per cent heavier than the standard plough. Trials show that the draught did not exceed 260 lb. as against the normal draught of 155 lb. of a standard plough. The

additional draught of the new plough is not likely to be heavy for bullocks as experience has shown that so far as ploughing is concerned bullocks are usually underloaded.

The quality of ploughing is also improved. Seasoned ploughmen who have used the new plough are enthusiastic about it and state that it is easier and less tiresome to operate due to its stability.

Besides its simplicity in construction, the new plough is comparatively economical, its cost being about 50 per cent more than the standard plough. It can be easily repaired and fabricated.

### Manufacture of Khandsari Sugar

THE IMPROVED PROCESS OF MANUFACTURE of *khandsari* sugar evolved at the Sugar Research & Testing Station, Bilari, is working successfully on a commercial scale in some of the private firms that have adopted it. The improved process gives a higher recovery of sugar than that attained in the indigenous open-pan process. The improved process gives a recovery of 7.92 per cent of cane as against 6.22 per cent by the indigenous process and the quality of sugar obtained is comparable with that of the ordinary vacuum-pan factory product.

The *Pheona Sugar Works*, Bijnore district, which has adopted the improved process, reports that the quality of sugar manufactured by this process is quite comparable with ordinary grade factory sugar and is selling at Rs. 28/8 per maund. The firm is willing to accord all facilities to intending *khandsari* sugar manufacturers to study the process at its factory premises.

### Indian Council of Agricultural Research, Annual Report, 1947-48

RICE, WHEAT, BARLEY, MAIZE, citrus fruits and commercial varieties of potatoes received special attention during the year 1947-48, according to the Annual Report of the Council, recently issued. On the veterinary side, bovine tuberculosis, rinderpest, foot and mouth diseases of cattle, nutritional deficiencies and Ranikhet disease of poultry were particularly investigated and useful results have been reported.

It has been shown that green manuring of paddy fields led to

higher yields than those obtained by keeping land fallow for a certain period. Growing *jowar* prior to paddy is injurious to the paddy crop; this practice should be abandoned. Research on new varieties of paddy showed that Chinese varieties gave a very heavy out-turn, viz. 60 to 70 md. per acre.

13 hill varieties of wheat were selected for tests on resistance to yellow rust. Varieties "23" and "34" were found resistant to yellow and black rust; 2 newly discovered strains "D" and "26" are resistant to brown rust.

Of the 19 research schemes on fruits, 8 were on citrus. Rootstock trials for *Sathgudi* orange and the common acid lime have been completed at Madras; the optimum season for budding has been determined. Rootgrafting of mango was successfully developed for the first time.

Over 200 potato hybrids approaching the common commercial varieties, both in tuber size and yield, were evolved at the Council's Potato Research Station at Simla. Some of the hybrids (2, 9, 12 and 19) are frost-resistant. The sprouting method of growing potatoes has been of immense value both in experimental and multiplication work.

The use of desiccated goat-tissue powder vaccine (D.G.T.) for rinderpest was tried on a field scale in the United Provinces. It was found that even under adverse conditions, the vaccine remained viable from 10 to 17 days, and it provided immunity to animals for a period of 7 months. Animals were also immunized employing the "Pill" vaccine, which consists of spleen pulp, 1 gm. (or 0.25 gm. of D.G.T. vaccine); glucose, 0.75 gm. and peptone, 0.1 gm. rolled up with glucose syrup or normal saline solution. These pills are not hygroscopic and completely diffuse in 45 min. Tests have shown that the pills retain their potency for a month when kept in a frigidaire and on inoculation produce reaction on the third or fourth day and confer a satisfactory immunity lasting for 17 months.

The comparative feeding values of straws of wheat, barley, *jowar*, *bajra* and paddy on milk production has been investigated in the United Provinces. It was found that the feeding value of paddy straw is superior to other straws. This is followed, in order, by

wheat, barley, *bajra* and *jowar* straws. It was also found that alkali-treated straw augmented milk production in cows.

On the nutritional side, experiments at the *Indian Veterinary Research Institute*, Muktesar, showed that the use of *vanaspathi* adversely influences the absorption of carotene, the absorption being 15 to 25 per cent less than that observed when ghee was used as the source of fat. *Vanaspathi* was also found to lower the absorption rate of calcium and phosphorus.

### Geodetic Work of the Survey of India, 1939-48

DURING THE LARGER PART OF the period, the geodetic activities of the *Survey of India* were concerned with the production of the war effort, according to a report prepared by the President, Survey Research Institute, Dehra Dun, and presented at the 8th General Meeting of the Geodetic Association held at Oslo in 1948.

From a review of the continuous chain of triangulation that exists from Syria to Malay, it has been possible to assess how much work remains to be done to continue the triangulation chain from India to Australia and Philippines, and to complete the block—Iran, Persia, India, Burma, Siam, French Indo-China and Malay.

Predictions of tides for an average of 39 ports between Suez and Singapore were carried out by the *Survey of India*. Proposals to increase the number of permanent observatories are being implemented, and a touring tidal detachment has started to take observations for a month in rotation, at older and secondary ports.

The Mineral Adviser to the Government of India in consultation with the *Geological Survey of India* had suggested some priority areas for gravity observations. Employing a Frost Gravimeter, a survey was carried out in the Raniganj coalfields, Bengal, and in an area north of Nagpur in Central Provinces during the field season 1947-48.

During the 2 field seasons, 1939-40 and 1940-41, 706 miles of high precision levelling in one direction was carried out and another 86 miles of high precision levelling and 252 miles of levelling in both directions have been added in 1946 and 1947, thus making a total addition of the

equivalent of 648 miles of completed levelling in both directions. The second net of levelling of high precision in India commenced in 1913-14, and this net when complete is expected to comprise about 15,800 miles. An important achievement has been the connection of the Indo-Burma levelling to that of the Siamese levelling by No. 2 Indian Field Survey Company in December 1945.

The total number of stations, latitude, longitude or azimuth at which the deviation of the vertical has been observed is now 1,162, at about 970 of which Hyford anomaly has been calculated. A volume giving these results is under preparation.

Geophysical prospecting was carried out in the Central Provinces with a view to test the applicability of certain geophysical methods for the systematic location of hidden ore bodies. Gradiometer and Walts vertical force variometers were employed to verify the geophysical indications in areas which had been already opened up. It was clearly established that both magnetic and gravimetric methods are suited for such surveys and the reef containing manganese ore bodies can be fairly accurately delineated.

### Indian Standards

THE *Indian Standards Institution* has brought out the following 8 Indian Standards which are now available to the public:

*Rules for Rounding off Numerical Values*—Indian Standard Rules for Rounding off Numerical Values, IS: 2-1949 (price as. 8). enunciates the problem that arises in rounding off of numbers which have to be expressed correct to several decimal places. The recommended rules for rounding off different classes of numbers, depending on the last integer to be rounded, and the explanation of the rules are covered by the Standard. The rules are so devised that subsequent operations performed on the rounded off numbers least affect the results.

*Inch-millimeter Conversion for Industrial Use*—For the use in industry, the ISI has published the Indian Standard on Inch-millimeter Conversion for Industrial Use, IS: 3-1949 (price as. 8). This Standard contains, in addition to the accepted ratio of conversion of inch to millimeters, conversion tables of inches to millimeters, millimeters to inches

and binary fractions of an inch to millimeters. Explanatory notes are appended to facilitate the use of the tables.

*Make-up of Periodicals*—The Indian Standard Practice for Make-up of Periodicals, IS: 4-1949 (price as. 8) provides an agreed set of rules for the lay-out of periodicals. The purposes of the rules is to enable editors and publishers so to shape the form of their periodicals as to facilitate their use by readers and librarians. The standard covers the requirements of individual issues, annual compilations and cumulative indices, and guiding rules to deal with abnormalities in the make-up of periodicals. The rules set out in the Standard are in general conformity with the recommendations of the *International Federation of Documentation*.

*Colours for Ready-mixed Paints*—The Indian Standard Colours for Ready-mixed Paints, IS: 5-1949 (price Rs. 4), corresponds to the 1948 edition of the B.S. 381C (93 colours) issued in March 1948 by the *British Standards Institution* as a revision of the 1930 edition. The *Indian Standards Institution* considered that in view of the wide usage in the past in India of the British Standard on the subject, it would be desirable to accept this standard for the present. Eventually an Indian Standard may be produced to cater to the particular needs of paint manufacturers and users in India.

*Fireclay Refractories*—The following 3 Standard Specifications for fireclay refractories have been drawn up for use in Indian industry:

- (i) Moderate Heat Duty Fireclay Refractories, Group 'A', IS: 6-1949 (Rs. 2);
- (ii) Moderate Heat Duty Fireclay Refractories, Group 'B', IS: 7-1949 (Rs. 1); and
- (iii) High Heat Duty Fireclay Refractories, IS: 8-1949 (Rs. 2).

These specifications cover the methods for chemical analysis, the determination of pyrometric cone equivalents under load for full-size bricks as well as for sections of bricks, the determination of porosity and resistance to spalling.

*Test for Shrinkage of Cloth on Washing*—The Indian Standard Method of Test for Determining Shrinkage in Woven Cotton and Linen Cloth in Washing, IS: 9-1949 (price as. 8) describes the

test for estimating shrinkage in cloth resulting from washing. The Standard gives the description of the washing machine, the test sample and the procedure for the estimation of shrinkage after laundering.

Orders for these 8 Indian Standards may be placed with the Secretary (Publications), *Indian Standards Institution*, Block 11, Old Secretariat, Delhi 2.

### Indian Standards for Nonferrous Metals

THE SECTIONAL COMMITTEE ON Non-Ferrous Metals of the Engineering Division Council of the ISI has prepared the following 16 draft Indian Standards to cover important non-ferrous metals used in Indian industry: Cast Aluminium Utensils; Wrought Aluminium Utensils; Zinc (Spelter); Pig Lead; Silver Solder; Soft Solder; Tin Ingot; Anti-friction Bearing Alloys; Copper; Phosphor Bronze Ingots and Castings; Brazing Metals; 98 per cent Aluminium Notched Bars and Ingots for remelting purposes; 99 per cent Aluminium Notched Bars and Ingots for remelting for Aircraft purposes; Aluminium Sheets and Coils for Aircraft purposes; Aluminium-coated High Tensile Aluminium Alloy Sheets and Coils for Aircraft purposes; and Aluminium-manganese Alloy Sheets and Coils.

Comments on these drafts (available with the Secretary, Publications) will be received by the Director, *Indian Standards Institution*, Block 11, Old Secretariat, Delhi 2, up to July 18, 1949.

### Indian Forest Statistics

A COMPREHENSIVE BROCHURE containing a large mass of statistical information regarding forest areas in India and their out-turn, imports and exports of various forest products, employment of labour in forestry and forestry industry, etc., during the period 1936-37 to 1947-48 has just been published by the Directorate of Economics and Statistics of the Ministry of Agriculture.

The data presented in the pamphlet mainly relate to Indian provinces, though some data in respect of a few States have been included, where available. The classification adopted in the

brochure is different from that prescribed by the F.A.O. in connection with the inventory of forest resources.

Among the items on which information is available in the brochure are: (1) forest area according to various classifications, e.g. ownership, reserved, protected, merchantable, unprofitable, timber producing, etc.; (2) areas showing progress in regeneration and afforestation; (3) areas under fire protection; (4) areas open and closed to grazing animals; (5) out-turn of timber and fuel according to different categories, namely timber, round wood, fire wood and charcoal; and (6) foreign trade in respect of wood and timber, wood products and minor forest produce. Sources of imports and destinations of exports have been given for the last three years.

### Narcotics Board for India

THE GOVERNMENT OF INDIA HAVE decided to set up an All-India Narcotics Board to improve and co-ordinate the various aspects of narcotics administration throughout the country and to implement India's obligations under international agreements.

The Board will consist of 6 members, comprising 2 representatives of the Central Government, 2 of the provincial governments and 2 of the states. The Board will employ a Narcotics Adviser, and the expenditure incurred by the Board will be met by income derived initially by the levy of a surcharge on the opium supplied to provinces and states.

The functions of the Board will be mainly to see that the various governmental understandings, agreements and obligations regarding narcotics administration are being observed and, in case of non-observance, suggest remedies to the authorities concerned.

To assist the U.N.O. in formulating a new Narcotics Limitation Convention, the Board will recommend to the International Control Board, through the Government of India, India's annual production and consumption quota. The Board will allocate among the various producing and consuming units within the country their production and consumption quotas as fixed by the International Control Board.

### Journal of the Zoological Society of India

THE FIRST NUMBER OF THIS *Journal* (half-yearly) contains 4 sections. The first section is devoted to original research paper and review articles, and the subsequent 3 to notes, reviews, recent advances and announcements. Among the articles included in this number are: (1) The fish fauna of the Rihand river and its zoogeographical significance; (2) On zoological standards and progress; (3) Life history and bionomics of the Potato Tuber Moth; (4) Notes on the free living and plant-parasitic Nematodes of Ceylon.

The *Journal* is well printed and neatly got-up.

### Announcements

*National Institute of Sciences of India*—At a meeting of the Institute held at Bangalore on 6th May 1949, the following awards were announced:

*National Institute of Sciences Senior Research Fellowship*—(1) Mr. U. R. Burman; (2) Dr. A. B. Kar; (3) Dr. S. M. Mukherje; (4) Dr. K. V. Srinath.

*National Institute of Sciences Junior Research Fellowship*—(1) Miss Ira Bose; (2) Mr. S. Datta Majumdar; (3) Dr. S. G. Joshi; (4) Mr. T. M. Mahadevan; (5) Mr. D. K. Mukherji; (6) Mr. K. Subramanyam; and (7) Mr. B. V. Sukhatme.

*Imperial Chemical Industries (India) Research Fellowship*—(1) Mr. A. K. Chakravarti; (2) Dr. A. P. Mahadevan; (3) Mr. C. Ramasastry; and (4) Dr. K. K. Reddi.

*UNESCO—Belgium Fellowship for India*—The Belgium Government has placed at the disposal of the UNESCO four fellowships to be offered, one each to China, India, Italy and Norway.

The fellowships are offered for advanced study in the following fields: *Education* (study of Decroly methods); *Museum* (problems and techniques of restoration of works of art), and *Fine Arts* (study of Flemish Art and the history of art in general).

These fellowships are intended for candidates already established in their professions for a period of six months, and carry a monthly stipend of 4,000 Belgian francs.

# Reports from States & Provinces

## HYDERABAD

### New Sugar Factory

COMPLETION OF A NEW PLANT AT the Bodhan Factory will make the combined unit the biggest factory in India. The present output of the factory is 120 tons of unrefined sugar and 3,500 gallons of power alcohol per day. When the new plant goes into operation, the daily output is expected to reach 300 tons of sugar and 9,000 gallons of alcohol.

The factory, which is 51 per cent State owned, is considered one of the most modern in India.

Extensive cane-breeding experiments are in progress at the research stations attached to the factory.

## MYSORE

### New Fertilizer Factory

THE GOVERNMENT HAVE CONCLUDED an agreement with the *Chemical Construction Co.*, New York, for the erection of a 50,000 ton fertilizer factory at Bhadravti at a cost of Rs. 2.5 crores. The factory is rated to produce 25,000 tons of ammonium sulphate and 25,000 tons of super phosphate per annum. The plant is expected to go into production in about 2 years.

## TRAVANCORE

### Jute Cultivation

THE TRIALS CONDUCTED IN Travancore with a view to put under jute cultivation 50,000 acres of low lying land have shown promising results. Within 2 months of plantation, the jute plants have grown to a height of 5' without any inter-cultivation or special manuring as is generally done in Bengal. In areas of low soil moisture content, seeds did not germinate, emphasizing the need for showers prior to the sowing of crop.

## WEST BENGAL

### Forest Development

THE FOREST DEVELOPMENT schemes in West Bengal envisage the conservation and improvement of existing private forests and the extension of forest areas by afforesting waste lands. At present, only about 15 per cent of the 28,000 sq. miles of the province is covered by forests.

The *West Bengal Private Forests Act, 1948*, provides for the conservation of private forests and afforestation of waste land. As long as private owners manage their forests according to the working plans approved by the regional Forest Officer, there is no interference by Government, but failure to do so enables Government to take over the management of forests as vested forests. At the controlled stage, Government demands no profit and technical service is provided free for the first 10 years. In the vested stage, Government's share of profit is 50 per cent. Afforestation by State is undertaken by agreement with private owners or by acquisition.

So far, 700 sq. miles of privately owned forests have been brought under the control of the State, and provision has been made to extend the *Act* over the entire province during the current year.

The Provincial Forest Advisory Board aims to utilize all available areas not suitable for production of food crops for afforestation, and the provincial Government has earmarked nearly the entire forest revenue for the conservation, development and expansion of provincial forests. State nurseries are being opened all over the province to offer facilities for tree-planting by villagers and afforestation of roadsides and waste lands.

An expansion of the existing coastal belt of tidal forests along the entire sea-coast of the province has been taken up and afforestation on an extensive scale has been planned for the current year. For this purpose, 19 ranges and 70 beats have been opened.

## BOMBAY

### Forestry College for Dharwar

A FORESTRY COLLEGE HAS BEEN opened in Dharwar—the first of its kind in the province and the third in India. The need for such a college arose because the existing facilities in the country for training of personnel in forestry are quite inadequate.

## MADRAS

### Lignite Deposits

DEPOSITS OF LIGNITE HAVE BEEN found extending over an area of 100 sq. miles between Vriddhachalam and Cuddalore areas, south of Madras city, during a survey carried out by Mr. H. K. Ghose, Lignite Officer, South Arcot. Preliminary surveys have disclosed that at several places in this area the lignite bed is about 80' thick. The seams dip seawards and thicken out in that direction. The lignite now found is of very good quality, the colour ranging from snuff to jet black. It can be used as fuel for industries, locomotive boilers, domestic purposes and for power generation.

## ERRATA

This Journal :

1949, Vol. 8, No. 5 : Page 164, line 30, article entitled "Stabilization of Plant Names", for "*Emolista*" read "*Emelista*".

1949, Vol. 8, No. 6 : Page 232, article entitled "A Study of the Colouring Matter of *Tambul* Seeds", the botanical name "*Zanthoxylum acanthopodium*" for "*Tambul*" was inadvertently omitted from the text.

# INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the *Gazette of India*, Part II, Section 1, for May 1949.

## Plastics & Plasticizers

38153. LEWIS BERGER & SONS LTD.: Manufacture of interpolymers of aromatic vinyl hydrocarbons and oils: *Heating with sulphur present in an amount 0.5 per cent to 5 per cent by weight of the drying oil which is present in an amount 10 per cent to 70 per cent by weight of reaction mixture.*

## Organic Chemicals

38677. I.C.I. LTD.: New azo dyestuffs: *Coupling diazotized 4-nitro-2-aminophenol-de-6-sulphonic acid with 1-amino-8-naphthol-3:6 di (sulphon-alkylamip).*

## Miscellaneous Chemicals

40066. FOUNDRY SERVICES LTD.: Heat producing mixtures: *Aluminium and an oxidizing agent characterized by the addition of metal fluoride.*

## Instruments (Professional, Scientific & Controlling)

39389. N. V. PHILIPS' GLOEILAMPENFABRIEKEN: Method of manufacturing magnetic anisotropic permanent magnets: *Alloy containing 30 to 42 per cent Co, 7 to 20 per cent Ni, 5 to 11 per cent Al, 2 to 10 per cent Ti, 0 to 10 per cent Cu, 0 to 2 per cent additional elements and the rest Fe and impurities, magnetized whilst cooling from a temperature exceeding the Curie point of the alloy down to a temperature 100°C. below it and annealed and finally magnetized, both magnetization being in the preferential direction.*

## Medical Research & Practice, including Clinical Application of Drugs & Pharmaceuticals

38573. DISTILLATION PRODUCTS INC.: Preparation of material having vitamin E activity: *Reacting beta or delta tocopherol to introduce methyl groups in the nucleus.*

## Metals & Metal Products

36182. MALHOTRA, PRAKASH & AGARWAL: Copper-silicon bronzes: *Preparing "hardner" alloy containing copper, silicon, manganese and iron, and adding the alloy to molten copper.*

41077. MOND NICKEL CO. LTD.: Cast iron: *Containing 2.0 to 4.5 per cent carbon, 0.1 to 3 per cent silicon, 0.1 to 2.0 per cent manganese, 0.15 to 0.5 per cent magnesium, 0 to 6.0 per cent nickel, 0 to 2.5 per cent chromium and 0 to 2.0 per cent molybdenum, the rest iron.*

## Rubber & Rubber Products

38489. DAYTON RUBBER: Method of making sponge rubber: *Mixing salt crystals with rubber-like*

*materials, vulcanizing the mixtures and removing the salts from the materials.*

38264. WINGFOOT CORPN.: Rubber hydrochloride film for wrapping packages: *Preparing film from rubber hydrochloride composition containing a hydrophilic surface active agent or treating rubber hydrochloride film with this agent.*

40721. WINGFOOT CORP.: Blended rubber latex frothed sponge: *Containing 70 per cent of rubber latex and 30 per cent of resin latex.*

## Textile & Textile Products

38426. DUNGLER: Autoclave for the continuous treatment of cloth at open width: *A close chamber, means for fluid-tight inlet and outlet of the cloth at open width, means for pre-impregnating the cloth with treating liquid.*

40755. NABAR & DARUWALA: A new method for the simultaneous dyeing of mineral khaki and vat dyes on textile materials: *The mineral salts, vat dyes and caustic soda being mixed together to form the dye liquor and the cloth subsequently subjected to the action of steam and hydrosulphite.*

## Food & Kindred Products

37377. GRIFFITHS HUGHES LTD.: Treatment of coffee beans: *Surrounding the coffee beans with waxes, heating the beans and finally removing the waxes from the body of the beans.*

## Packing & Packaging

40551. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ: Process for the treatment of materials, particularly packing material, so as to cause said material to emit corrosion-inhibiting vapours: *Applying inorganic nitrite and salt of organic base on packing material and crystallizing nitrite of organic base on said material.*

38637. PRASAD & PRASAD: Packing and preservation of fruits, vegetables and the like: *Packing material comprising chemical preservative, saw dust and/or wood shavings and/or magnesium carbonate.*

## Miscellaneous

39486. SOCIETE INTERNATIONALE SOIE-COSMETIQUE: Method for pulverizing fibres of animal and vegetable tissues: *Subjected to oxidation and then ground.*

39661. CHAUDARY: An improved filter-press: *Upper compartment of cylinder has perforated cylinder with conical opening and lower compartment has a cone for removal of cake formed in perforated cylinder.*



# Need for Research on Malaria Chemotherapy in India\*

SOME idea of the magnitude of the malaria problem in India may be obtained from the statement of Hehir who estimated in 1927 that there were about 100,00,000 cases of malaria occurring annually in India. During war-time, the incidence of the disease must have assumed more serious proportions because of the unchecked growth of the malaria-carrying mosquitoes from untreated swampy areas, their rapid transmission from place to place and the acute shortage of proper drugs to treat "carrier" cases. The total number of malaria cases in India must have, therefore, increased very significantly during the period from 1927 to to-date. The annual economic loss from malaria alone is consequently colossal, and it is believed in very authoritative quarters that if proper *curative* treatment could be elaborated for this single disease, India can be converted in a single generation to one of the most prosperous and healthy countries of the world.

## Fundamental Objective of Malaria Chemotherapy

The objective of our research in this field is the control of malaria by *chemotherapy* or *chemo-prophylaxis*. There is a growing volume of opinion that the only effective way through which malaria can be *radically eradicated* is through the discovery of a drug or drugs capable of *sterilizing* the patients from the malarial infection. Preventive methods, through anti-mosquito campaigns and other methods of larvae control which have been practised widely during the last half a century, though useful, have been proved in many parts of the world to be *ineffective per se*. That we have fallen far short of our goal of *total eradication* of

malaria in spite of all types of anti-malarial measures is only too evident to need further emphasis. According to Coggeshall of the Rockefeller Institute for Medical Research, New York, malaria as a world disease is probably on the increase at the present time.

This objective may be attained in 4 ways :

(i) Determination of the optimum method of using known anti-malarial drugs (e.g. quinine, mepacrine, etc.) in "suppressive" treatment ;

(ii) discovery of drugs which can act as true causal prophylactics (e.g. sulphadiazine in gallinaceum malaria in the chick) in human malarias and which can be given to man with safety over extended periods of time ;

(iii) discovery of drugs which will consistently eradicate the parasites in human infections and thus prevent relapses which commonly occur after "clinical cure" with quinine or mepacrine ; and

(iv) discovery of drugs which have anti-malarial properties qualitatively similar to quinine or mepacrine, but which possess a larger margin between the effective and toxic doses.

The first item is more or less thoroughly worked out as a result of studies made both in Great Britain and America during the war (1941-45). A good review of work already completed in this field is available from a paper by J. A. Shannon and others.\* The other three ways of attaining the objective have also been intensively pursued, particularly, comprehensively in America. An extensive programme of research, supported mainly by the Committee on Medical Research (CMR) of the Office of Scientific

\*J. Phar., 1944, 81, 3730.

\* From a scheme submitted before the Drugs & Pharmaceuticals Committee of the Board of Scientific & Industrial Research (Chairman — Dr. Jivraj N. Mehta).



Research and Developments (OSRD), was launched as early as 1941 with a view to stamp out the dread of malaria from the fighting forces in the tropical theatres of the last global war. A detailed account of this American scheme on anti-malarial research which involved the co-operation with civilian scientists, scientists from the U.S. Army, Navy and Public Health Services, workers in industrial firms and also international co-operation with such countries as China, Great Britain, Canada, Australia, Argentina, French Guinea, Columbia, Cuba, etc., has now been published in the form of a monograph entitled *Survey of Anti-malarial Drugs* (2 volumes), edited by F. Wiselogle. This report indicates that in the search for newer anti-malarials other than the cinchonas, no significant progress can be made unless a close co-ordination can be established between the synthesis of new compounds, pharmacological and experimental therapeutic investigation of these compounds in animals, and clinical trial in human malaria of selected compounds under carefully controlled conditions.

#### Present Status of the Problem

The subject is so vast that any attempt made to summarize the picture in a short compass would result in more confusion than clarification of the problem. In brief, it may be stated that in spite of the very valuable researches in the field of malarial chemotherapy so far reported, an *ideal anti-malarial drug* has not yet been found and, therefore, there is room for further search. Further, most of the findings on which our present concepts on the use of such drugs as chloroquine and paludrine are based are dependent on experimental data on strains of malaria parasites of American, Australian and south Pacific origin, which may not necessarily be applicable to Indian strains of malaria; at least this has to be confirmed experimentally in India.

#### How Success Can Be Achieved?

Complete success in the elaboration of a truly effective anti-malarial would involve:

- (i) An improvement in suppressive therapy (this has been achieved to a certain extent by the discovery of chloroquine and paludrine);
- (ii) an improvement in the treatment of clinical attack (also partially achieved);
- (iii) the development of agents which would cure *falciparum* malaria (now achieved)

and *vivax* malaria at a well-tolerated dosage; and

(iv) finally, the development of agents which would prevent the inception of these diseases (true causal prophylactic).

#### Target in Anti-malarial Chemotherapy

The target to be arrived at can be briefly stated as follows:

(a) The discovery of a true causal prophylactic for human malaria—a drug which would eradicate some stage of the parasite before trophozoites were produced—would constitute a great advance.

(b) The discovery of a drug which would resemble mepacrine in its anti-malarial properties but which, in addition, would cure *vivax* malaria. This would probably be of more value than (a) (e.g. sulphadiazine in chick malaria which is a prophylactic but not a curative drug).

(c) Another ideal approach would be through the use of drugs hampering the activity of the enzyme system or metabolites essential to the malarial parasites but not necessarily so to the host (e.g. activity of sulpha drugs through p-aminobenzoic acid system).

The “leads” that suggest themselves to research workers from a critical perusal of anti-malarial drug research so far conducted are given in the Appendix at the end.\*

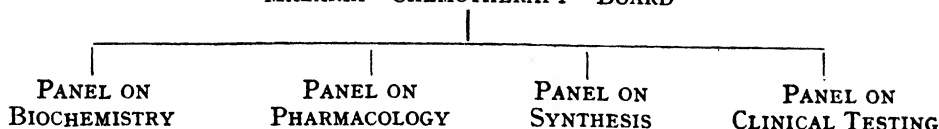
#### Co-ordination of Malaria Study in India

In view of the supreme importance to India of research in malarial chemotherapy and in view of what has been done in countries where the malaria problem is of far less magnitude than in our country, it is very desirable that immediate steps be taken for the initiation and active continuance of chemotherapeutic researches with special reference to malaria. As indicated in the Appendix, a number of useful “leads” are already available and this can be pursued provided an organization more or less on the lines adopted by the “Board for the Co-ordination of Malaria Studies” of the United States of America is set up in India. The set-up of the unit established in America during the war period under the auspices of the Office of Scientific Research and Development is shown in the chart.

The Board consisted of a President who was helped in his work by a Technical Executive Secretary and a Technical Aide, the latter maintaining constant liaison with the

\* Marshall, *Federation Proceedings*, 1946, 5, 298.

## MALARIA CHEMOTHERAPY BOARD



chairmen of the 4 panels. Each panel had, in addition to a chairman, at least 3 members acknowledged to be experts in their respective fields. The chairman of each panel was responsible for initiating work but the members had an independent status and had full freedom to suggest problems and undertake work after the programme had been discussed with the chairman. Each panel was provided with a technical secretary, who undertook the review of progress of work in his field all over the world. Financial support was made available to this body from the Office of Scientific Research and Development and many private agencies also collaborated with men and money to make the effort successful.

If it is not possible to set up as elaborate an organization as planned in America, there is good reason to feel that the time is at least opportune for the setting up of a broad-based committee of experts who could probe into the problem of chemotherapy of malaria with the full knowledge of the complexity of the Indian problem and suggest solutions likely to be of value to the therapy of Indian strains of malaria. The Malaria Institute of India is already tackling the Indian malaria problem with commendable zeal and energy. It is for the chemotherapeutists of India to come forward and help the Institute with newer synthetics for their studies, or take their advice for further development in this direction.

For any success of this scheme one thing must be emphasized and that is that this kind of research involves the contribution of many sciences, and, therefore, "team work" is essential for rapid progress.\* The

\* The principle of collaborative work between all concerned bodies on malaria research in India was approved by the Drugs & Pharmaceuticals Committee and later by the *Board of Scientific & Industrial Research* at their meeting held on 18th January 1949. The *Board* recommended that a standing committee for malaria chemotherapy be constituted to co-ordinate and direct research and other investigations in the subject carried out in India. This committee consists of 8 members excluding the chairman, the personnel being drawn as follows:

1. Chairman of the Pharmaceuticals & Drugs Committee of the *Board of Scientific & Industrial Research* or his nominee.

chairman of any such committee must be a scientist of wide and comprehensive outlook with a *drive* which will make working in such a team both pleasant and profitable. The potentiality of individual thought and work upon the numerous fundamental problems of malaria must be appreciated and at the same time centralization and team work emphasized. Research in this field holds great promise for the future and there is no reason why an Indian should not be competent to contribute to this programme which has already engaged international attention. Even if no tangible results are arrived at from Indian work, the training which the workers will get from such a collaborative research undertaking will pave the way for some unknown achievement in the synthetic and chemotherapeutic field.

## APPENDIX \*

"Leads" from Anti-malarial  
Drug Research so far Conducted

(a) 4-Amino-quinolines — If one takes the formula (FIG. 1) for mepacrine and removes either of the outer rings, one obtains two 4-amino-quinolines, one with a methoxy group in the 6-position and one with a chlorine group in the 7-position. The first of these compounds belongs to a series which, in its anti-malarial properties, resemble quinine and mepacrine rather than the 8-amino-quinoline, pamaquine.

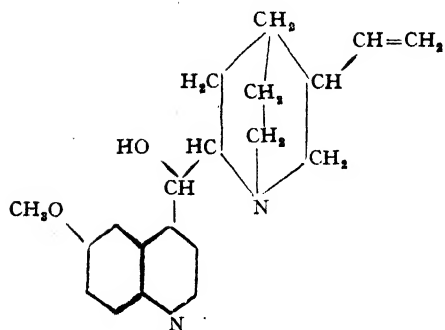
After examining nearly 200 compounds in this series, the American workers have screened 10 of these 4-amino-quinolines, which appear to be superior to mepacrine. Chloroquine (SN 7,618) has received the most extensive study in both

2. Six members (from various learned bodies, research institutions, defence science or medical organization, Malaria Institute, etc.) to be nominated by the chairman.

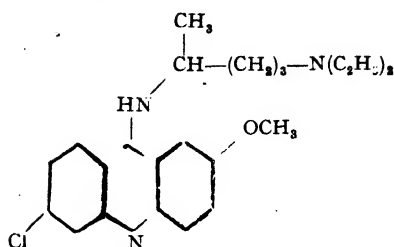
3. Two members to be nominated by the Scientific Advisory Board of the Indian Research Fund Association.

The Secretary of the Drugs & Pharmaceutical Committee was nominated as the Secretary of the Malaria Chemotherapy Committee.

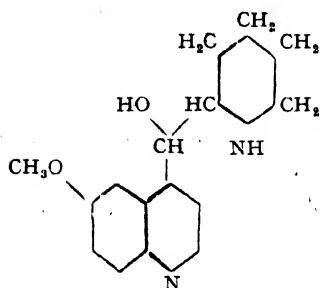
\* This statement takes note of only the highlights of chemotherapeutic research on anti-malarials and is not a comprehensive review of the vast field already covered in this field.



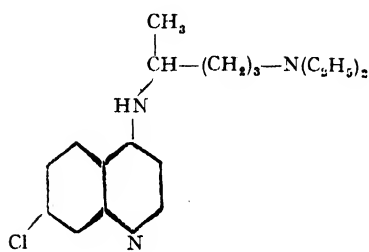
## QUININE



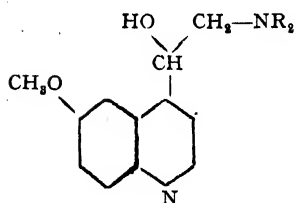
## QUINACRINE



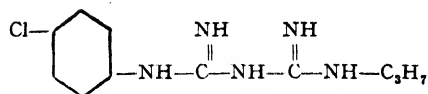
AINLEY-KING TYPE



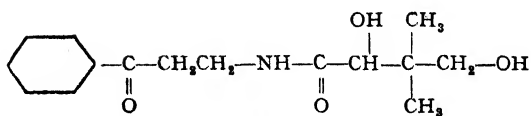
CHLOROQUINE (SN 7,618)



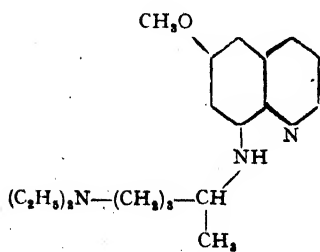
**KING-WORK TYPE**



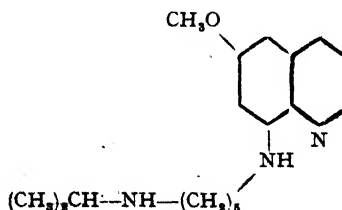
### CHLORGUANIDE (PALUDRINE)



PANTOTHENOPHENONE (SN 12,610)



## PAMAQUINE



PENTAQUINE ( SN 13,276 )

FIG. 1 — FORMULAE OF ANTI-MALARIAL DRUGS.

civilian and military establishments. It is superior to mepacrine in a number of ways. Effective suppression can be obtained by administering it no more frequently than once a week in a well-tolerated dose. It will cause an abrupt termination of the clinical attack of *vivax* malaria and will cure *falciparum* malaria when administered for only one or two days. In addition, it does not stay in the skin or produce gastro-intestinal disturbances. In this field further work is urgently called for. It would, for example, be necessary to know the nature of the metabolic degradation products of 4-aminoquinoline derivatives (chloroquine) in human beings. Some knowledge about the metabolic degradation products of quinacrine has now been obtained from biochemical studies of excretory products from human urine. If similar data on chloroquine, etc., are obtained, suitable substituted alkylamino and/or more stable nuclear-substituted compounds could be synthesized in that series.

(b) *8-Amino-quinolines* — It is now practically certain that quinine and pamaquine, under certain conditions, will cure both American and South-west Pacific strains of *vivax* malaria. However, there is probably not a sufficient margin between minimal curative and maximum tolerated doses to warrant its use at this time. About 20 of the 8-amino-quinolines have been tried for their curative effects in *vivax* malaria. One compound, SN 13,276 (pentaquine), appears to be curative in a smaller dosage than pamaquine and to be about one half as toxic for man. Possibly this drug or similar drugs in this series will have a wide enough margin of safety to be used in the cure of *vivax* malaria. As the main objective would be to search for a true prophylactic, an intensive investigation in this series appears more promising as these are the derivatives which have shown gametocidal activity also.

(c) *Quinoline-methanols* — These compounds may be considered as being rather closely related to quinine. Ainley and King in 1938 reported the first compound of this series to be found active in avian-malaria. Later, King and Work prepared a series (see formula) and found them also to possess anti-malarial activities. About 200 compounds of the quinoline-methanol type have been prepared and in both the A-K and K-W series, we have now compounds about 40 times as active as quinine. About 5 compounds of this

series have been tried in man for anti-malarial activity and comparatively high toxicity. However, due to poor absorption and toxicity, none of these compounds have appeared promising. Recent findings have shown that quinoline ring is not necessary for anti-malarial activity and it would, therefore, be of interest to introduce newer chain or nucleus to these methanol derivatives.

(d) *Pantothenic Acid & Analogues* — Trager\* reported that the survival *in vitro* of *P. lophurae* was favoured by the presence of calcium pantothenate. This observation resulted in the synthesis of about 25 analogues of pantothenic acid for anti-malarial testing. A compound synthesized by Woolley† and found by him to be an antagonist for pantothenic acid in the case of bacteria, was found, when given orally, to be as active as quinine in *gallinaceum* malaria in the chick, active in *lophurae* malaria in the chick, but inactive in *lophurae* malaria in the duck. This substance, pantothenophenone, SN 12,610, was found definitely active in *vivax* malaria in the human being. This is the first instance in which an effective anti-malarial agent has been found as a result of a logical, rather than a "hit or miss" approach. However, much success has not been achieved in the parallel field of sulpha drugs therapy by working on this plan. The bottle-neck lies in our rather imperfect knowledge of the nutritional requirements of malaria parasites and their metabolic peculiarities.

(e) *Guanides, Amino Phenols, etc.* — The discovery of chlorguanides (paludrine) has switched the attention of chemotherapeutists to another new angle altogether. Though chlorguanide is "prophylactic" in *falciparum* malaria and partially so in *vivax* malaria, it is usually considered useful only as a "suppressive" drug. It may be possible, through further studies, to develop simple basic compounds such as other guanides, amino acid derivatives, amino phenols, etc., that might react with the trace elements or complex pyridine bodies like pyridoxine resulting in the elaboration of newer synthetic derivatives of value, both in prophylaxis as well as in suppression and cure of malaria. This field still remains largely unexplored.

B. MUKHERJI

\* *J. Exper. Med.*, 1943, 77, 411.

† *Sci.*, 1944, 100, 579.

## Board of Scientific & Industrial Research

### Twenty-third Meeting, New Delhi

**T**HE twenty-third meeting of the *Board of Scientific & Industrial Research* was held in New Delhi on July 11, 1949. The Hon'ble Pandit Jawaharlal Nehru presided.

On the recommendation of the Board, the Governing Body of the *Council of Scientific & Industrial Research* sanctioned 13 new schemes :

1. Synthesis of citrinin and preparation of derivatives of citrinin — DR. K. VENKATARAMAN (*Bombay*).

2. Elucidation of the constitution of essential oils and their synthesis — DR. P. C. GUHA (*Bangalore*).

3. Physico-chemical properties of hides — tanned and untanned — DR. B. N. GHOSH (*Calcutta*).

4. Scheme of research on leather — MR. B. M. DAS (*Calcutta*).

5. Studies on colloidal instability of cloud particles above and below the freezing temperature — DR. S. K. BANERJEE and DR. L. S. MATHUR (*New Delhi*).

6. Polarization of down-coming radio waves — DR. S. R. KHASTAGIR (*Banaras*).

7. Cellulose decomposition by soil fungi with special reference to its application in fermentation technology — DR. T. S. SADASIVAN (*Madras*).

8. Enzyme bates—MR. S.N. Sen (*Calcutta*).

9. Research on soft X-rays — PROF. S. N. BOSE (*Calcutta*).

10. Development of magnetic dust cores — MR. B. D. TOSHNIWAL (*Bombay*).

11. Development of an electronic type of frequency analyser — DR. N. B. BHATT (*Bangalore*).

12. Study of intensity variation of short-wave radio signals and their bearing on the ionosphere — DR. S. S. BANERJEE (*Banaras*).

13. Studies of correlation between abnormal variation of region of ionization of the ionosphere and similar variation of the night sky emission — DR. S. N. GHOSH (*Calcutta*).

The setting up of a pharmacological unit, temporarily at Calcutta, under the direction of Dr. B. Mukherji was approved. This unit will be transferred to the Central Drug Research Unit, Lucknow, as soon as its laboratories are fully equipped and start functioning.

The Central Drug Research Institute will have a full division for undertaking botanical research on indigenous drugs. This division, in addition to carrying out laboratory investigations, will undertake experimental cultivation, surveys, grading and sorting of indigenous drugs and also serve as a centre of intelligence and technical assistance to the indigenous drug industry.

The Governing Body of the Council considered the report of the Reviewing Committee which was appointed during 1947 to review the utilitarian aspects of the Council's activities. This Committee (Chairman : Mr. Ardeshir Dalal) undertook a survey of the activities of the Council and the findings are that the *Council of Scientific & Industrial Research* has done much valuable work during the short period of its existence. Some of the recommendations of the Committee with regard to increasing Council's usefulness to industry are greater liaison with industry, improvement in the publicity organization of the Council, setting up of pilot plants in the National Research Laboratories, and the establishment of a Board of Engineering Research under the auspices of the Council.

In order to facilitate a better and more complete utilization of the results of research in the country, the *Council of Scientific & Industrial Research* has recommended that the Government of India should set up a National Research Development Corporation on the lines of similar organizations in America, Britain and Canada. The primary object of the corporation will be to conduct semi-commercial trials of new processes developed in India and thereby encourage industry to adopt them in large-scale practice.

It has been recommended that the corporation should be floated with a capital of Rs. 50 lakhs and a recurring expenditure of Rs. 5 lakhs to start with, and the co-operation of all State-owned and State-controlled industries and of private concerns would be sought in the efficient working of the corporation. A committee with Mr. Ardeshir Dalal as the chairman, Dr. J. C. Ghosh, Mr. Kasturbhai Lalbhai, Mr. Sri Ram and Dr. S. S. Bhatnagar as members has been set up to work out the details.

The Governing Body approved the installation of pilot plants in all the National Research Laboratories. A programme has been outlined to render increased technical assistance to industry. An Industrial Relations Officer is being appointed to maintain liaison with all the major trade associations and chambers of commerce. Technical personnel of the Council will visit industrial establishments to render such technical aid and advice as may be necessary for the exploitation of new processes.

A Board of Engineering Research is to be established under the auspices of the *Council of Scientific & Industrial Research*. The primary function of this body will be to co-ordinate research work carried out in various engineering establishments under the Government of India.

With regard to the possibility of producing synthetic petrol from coal, the Council has proposed that pilot-plant investigations should be conducted in India itself so that all types of low-grade coals obtaining in India can be examined. Two other proposals put forward relate to the utilization of pulverized coal for firing stationary and locomotive boilers and the development of a turbine for locomotives employing pulverized coal. As an experimental measure, the Council has suggested importing a boiler in which pulverized coal can be employed. The unit is to be set up at one of the thermal stations in the Damodar Valley area.

A Fuel Economy and Combustion Section is being organized in the Fuel Research Institute, Dhanbad, to advise consumers of coal on coal conservation and economizing methods.

At the instance of the Council, the Indian Standards Institution has constituted a committee to fix standards of purity for salt manufactured in India for various purposes, and the Council has recommended that these standards be soon introduced in the salt industry. A Salt Research Station is to be established at Wadala, Bombay, where scientific methods of salt production would be worked out and demonstrated to salt producers. It is also proposed to establish another research unit at Sambhar Lake area to deal with the problems of salt manufacture from lake and pit brine. The Council has recommended to the Government of India that the Mandi salt mines and the salt deposits in the Rann of Cutch should be developed.

The proposal of the Indian Statistical Institute for the establishment of a research unit in quality control to serve the needs of industries in Bombay has been accepted by the Council. This research centre will be attached to training centre in quality control financed by industry in Bombay.

The Council approved the appointment of Professor Kriedel, Director of Research, *Bausch & Lomb*, U.S.A., as Director, Central Glass & Ceramic Research Institute, Calcutta.

## Characteristics of the Ionosphere over Calcutta (May 1949)

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(Miss) T. K. PAL & A. P. MITRA

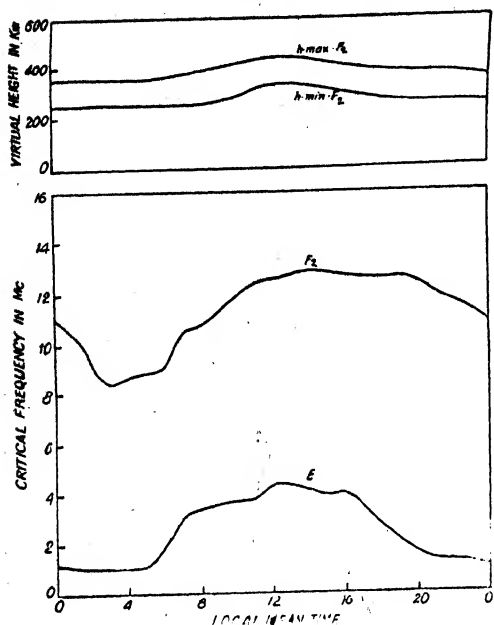
*Wireless Laboratory, University College of Science, Calcutta*

THE following are the ionospheric data observed at Calcutta for the month of May 1949. The observations were made at each hour of the day for 5 days a week.

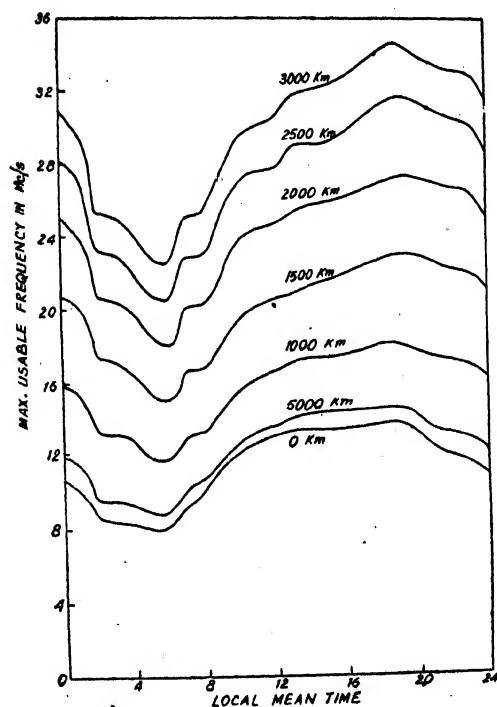
Fig. 1 represents the mean hourly values of the virtual heights and critical frequencies of the  $F_2$  layer and the critical frequency of the E layer in graphical form during the month of May 1949. Fig. 2 gives the

predictions of maximum usable frequencies which can be used for different distances of transmission during August 1949 by reflection at the F region over Calcutta. Table I gives the list of occasions when E region ionization was found to be abnormal and the corresponding penetration frequencies and virtual heights.

There were frequent occurrence of thunderclouds during this month at different times



( 5 HOURS 54 MINUTES AHEAD OF G.M.T. )  
FIG. 1 — MAY 1949.



AT POINT OF REFLECTION.  
( 5 HOURS 54 MINUTES AHEAD OF G.M.T. )  
FIG. 2 — PREDICTED M.U.F. VIA  $F_2$  LAYER,  
AUGUST 1949.

of the day and the E region ionization was found to be abnormal on most of the occasions. The afternoon abnormality in the F region ionization observed during the previous months has almost disappeared in the month of May. During the last few days of the month, however, the ionosphere was found to be disturbed.

TABLE I

MONTH & YEAR	DATE	HOURL	$f_o^o E_s$ Mc.	$h E_s$ Km.
May 1949	2	16.00	4.35	135
		17.00	3.50	135
		18.00	3.30	120
	3	20.00	3.25	120
		21.00	3.00	120
		22.00	2.70	105
	*5	23.00	2.00	105
		10.00	5.00	135
		11.00	5.20	135
	*10	12.00	5.00	135
		16.00	4.65	135
		17.00	4.20	135
	*12	18.00	6.80	135
		19.00	5.50	135
		20.00	5.35	135
	*13	21.00	5.30	135
		22.00	5.05	135
		19.00	7.05	135
	13	20.00	7.00	135
		21.00	6.00	135
		22.00	3.50	120
	13	23.00	2.90	120
		00.00	3.80	105
		01.00	3.00	105
	*16	02.00	2.75	105
		05.00	3.50	105
		16.00	4.75	120
	18	17.00	4.80	120
		18.00	4.80	120
		07.00	4.10	120
	*19	08.00	4.75	120
		20.00	4.25	120
		21.00	4.20	120
	20	22.00	4.30	120
		23.00	4.25	120
		*00.00	4.00	105
	21	07.00	7.25	120
		08.00	7.45	135
		09.00	7.70	135
	23	19.00	4.75	150
		*20.00	4.75	150
		21.00	4.00	135
	24	23.00	3.75	135
		00.00	3.00	120
		17.00	3.15	120
	25	18.00	3.05	120
		23.00	2.80	105
		00.00	2.70	90
	*26	01.00	2.70	90
		17.00	6.00	135
		*18.00	3.65	135
	27	*21.00	3.25	135
		09.00	4.50	135
		10.00	6.00	150
	*30	11.00	10.70	150
		12.00	11.00	135
		18.00	3.55	135
	31	19.00	6.75	135
		20.00	5.75	135
		21.00	5.85	135
	31	22.00	6.20	135
		00.00	3.55	120
		01.00	3.05	105
	*30	02.00	2.70	90
		03.00	2.35	90
		14.00	7.45	150
	31	15.00	7.50	150
		16.00	7.90	150
		17.00	7.50	150
	31	22.00	3.00	105
		23.00	2.55	90

\* Local thunder-storms

# The A.C. Network Analyser

M. S. THACKER

*Department of Power Engineering & Electrical Technology,  
Indian Institute of Science, Bangalore*

**T**HE past decade has witnessed a very rapid increase in power development and power systems in the Western Hemisphere. India is on the threshold of a vast development of her potential power resources — its best, efficient, and economic utilization by an all-India interconnection of power systems — in the creation of a "National Grid".

In this approach to power development, numerous problems, both engineering and scientific, have to be investigated and solved. Many investigations involved in this connection would be difficult to undertake because the amount of work and time required to arrive at satisfactory quantitative conclusions would be prohibitive. This fact has been more emphasized because of the necessity for rapid technological progress over a period of time during which there would be a relative dearth of technically trained men. The engineer or the scientist, fully aware of this situation and of the need for theoretical understanding in quantitative terms, has to look to ways and means for overcoming this limitation of man-power and also of time.

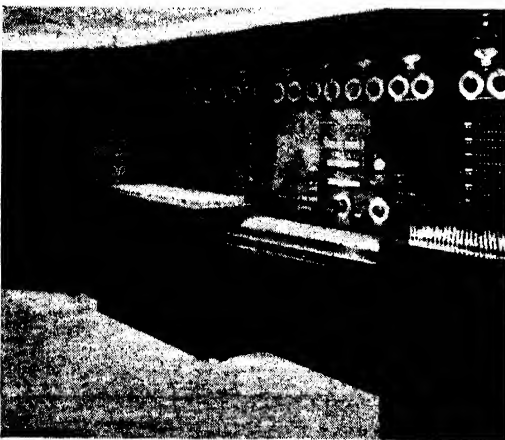


FIG. 1 — THE A.C. NETWORK ANALYSER, INCORPORATING INDUCTANCE AND CAPACITANCE IN ADDITION TO RESISTANCE IN ITS IMPEDANCE UNITS, IS APPLICABLE TO A WIDE RANGE OF DESIGN AND OPERATION PROBLEMS.

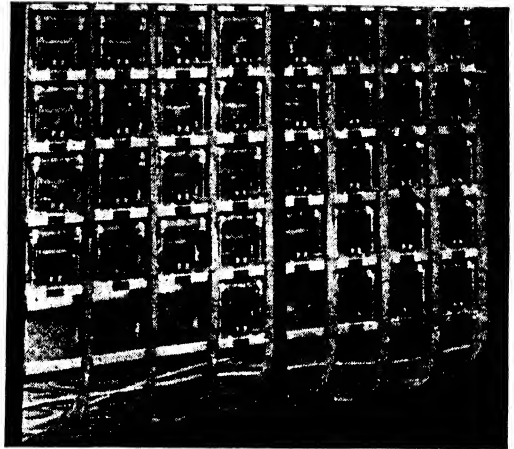


FIG. 2 — REAR VIEW OF TWO CIRCUIT-UNIT CABINETS SHOWING THE WIRING DETAILS.

For studying more readily the numerous problems associated with power system planning, design and operation, experimental methods of solution have been evolved and apparatus for such studies is built, commonly referred to as "Network Analyser" or "Network Calculator". The use of such an analyser will be invaluable for national power planning.

A general view of this analyser board is shown in Figs. 1 and 2. It consists essentially of numerous adjustable impedance units of inductance, resistance and capacitance, such that the power systems or other electrical networks can be represented by them. Generators and impedance units can be set up to represent the desired system or systems and equivalent circuits. The system studies can be made fast, flexible and accurate. Representative circuits set up would efficiently solve certain problems which cannot otherwise be practically handled.

The types of information obtainable from the A.C. network analyser is indicated below. It is classified according to four general types of problems for which the analyser is particularly adopted, but which would suggest other uses. The list below is not final



or complete but is merely indicative as a guide.

### **Power System Planning, Design & Operation Problems**

#### *Load Division —*

- Desirable and economic location for new generation
- Effect of adding new transmission circuits
- Determination of location for new sub-station
- Effect of bus reactors
- Effect of increased system loadings
- Effect of changes in transformer reactance
- Effect on system of temporary loss of various transmission circuits
- Effect of temporary loss of generation or synchronous-condenser capacity
- Circuit loadings
- Bus voltages
- Reactive-power capacity needed
- Ratings of transformers and transformer windings
- Tap range of transformer required
- Benefits from load ratio control
- Benefits possible through use of phase-shifting transformers
- Effect of increased system voltages
- Effect of increased conductor sizes
- Determination of reactive-power flow; and
- Investigation of system losses and means of reducing them

#### *Short-circuit, Three-phase & Unbalanced Faults —*

- Maximum short-circuit currents for determining circuit-breaker duties
- Bus voltage during short circuits as a measure of fault severity and net effect on system
- Maximum and minimum fault currents and voltages for determining relay settings
- Determination of zero-sequence currents for inductive co-ordination problems
- Effect of mutual coupling between circuits on short-circuit currents and voltages
- Determination of grounding-transformer rating
- Effect of ground-fault neutralizer on fault currents in the transmission network
- Maximum and minimum fault currents for application of expulsion protective tubes; and
- Effect of various types of system grounding

#### *Stability —*

- Steady-state limits
- Transient limits with various types of faults;
- Switching times required for various types of faults and fault locations
- Effect of bus reactors on transient stability
- Effect of  $Wk^2$  on transient-stability limits
- Effect of transient reactance on transient-stability limits
- Determination of increase in transient-stability limits with shorter switching and relay times
- Effect of switching and of bus arrangements on stability limits
- Effect of quick reclosure of circuit breakers
- Extent to which transient-stability limits are affected by saliency and excitation response
- Effect of damping torques on transient-stability limits
- Time required for generator to pull out of step after loss of excitation, and effect on the rest of the system; and
- Evaluation of effect of single-phase switching and reclosing on transient-stability limits

#### *Special Circuit —*

- Starting and pull-in characteristics required for large synchronous motors connected to a transmission system
- Determination of desirable methods of starting large synchronous motors or synchronous condensers
- Reduction of a complicated network to a more simple equivalent
- Determination of currents and voltages in three-phase circuits containing unbalanced impedances
- Cable-sheath currents
- Determination of fifth-harmonic voltages generated in a system by transformer magnetizing currents
- Solution of equivalent circuits representing mechanical elastic, and dynamic systems
- Solution of equivalent circuits for the hunting of electric machinery
- Determination of characteristics and performance of variable-ratio frequency-changer sets; and
- Solution of equivalent circuits representing field equations of mathematical physics

Apart from the applicability of the analyser to power system problems, some of which are briefly enumerated above, there are several significant special scientific applications which point to a wide field of the analyser's usefulness.

Among these applications may be mentioned the compressible-fluid-flow problem for either subsonic or supersonic flow; the equivalent circuit for an elastic field; equivalent circuits for elastic structures; study of the propagation of electro-magnetic waves; the Schrödinger amplitude equation for one, two and three independent variables in orthogonal curvilinear co-ordinate systems; study of certain mechanical vibration problems; hunting in certain electro-mechanical systems; the process of "scanning" the hunting network; solution of hunting problems associated with power selsyns; solution of the problems of doubly fed machines; developments of equivalent circuits for electrical machinery for hunting and unbalanced operation, etc.

The well-known analogies among mechanical, electrical, acoustic, thermal, and other systems would suggest many possibilities. If researches could be undertaken and the complete science of hydraulics be built up into equivalent electrical analogies, the possibilities of adapting the analyser to solutions of problems over the entire field of power engineering would be great.

The network analyser, which is being installed in the Power Engineering Department at the *Indian Institute of Science*, and which is expected to be ready to function by early 1950, consists of a power supply system embodying 16 generator units, each consisting of 2 power selsyns for independent phase-angle and voltage magnitude adjustments; 16 synchronous impedance units for use with the generators to represent synchronous impedances of generators in actual system; 100 line units consisting of adjustable resistors and reactors, intended for representing lines or other circuits of relatively small impedance; 50 load units also consisting of adjustable resistors and reactors which may be connected either in series or in parallel (the impedances of these units are much higher than those of the line units); 48 capacitor units to represent line capacity, synchronous condensers,

or low valves of negative reactance; 32 variable ratio auto-transformer units for stepping up or down the voltage at different points in the network; 8 mutual reactor transformer units with 1:1 ratio used in conjunction with a line unit to represent the mutual impedance of parallel circuits.

In addition, there are jumper circuits to serve as zero-impedance ties between points in the network or as connectors for metering purposes. They are useful to represent bus-tie breakers, or the terminals of equivalent "pi" transmission lines, tie different phase-sequence networks together during certain types of fault studies to totalize currents of two or more circuits connected to a bus, and any other purpose that requires a zero-impedance tie.

The A.C. network analyser will solve efficiently many problems which cannot be handled practically by any other means. It is hoped, the analyser will be constantly in use by the Central, Provincial and State Electricity Planning Commissions, Boards and authorities, and also by private electricity authorities and industries. Such network analysers in the U.S.A. and in the U.K. are kept in constant operation, even working on three shifts a day, for the solution of various problems in the power system development in these countries.

There is another type of analyser developed for studies, such as the power system electric transient phenomena. This is the transient network analyser and permits studies of problems involving switching with restriking, transmission-line or similar distributed-constant systems, non-linear impedances such as saturable reactors and non-linear resistors, and circuits involving commutation or discontinuities such as rectifiers and inverters. It is a unique calculating device for numerous problems involving distributed constants, non-linearities and discontinuities which it can handle in either the transient or steady state readily and quickly. The problem need not be an electrical one primarily, but may be any problem which can be reduced to an appropriate electrical equivalent. This type of analyser is not of immediate necessity, but its installation in not too distant a future is visualized.

# Practical Working of a Laboratory Equipment for Thermionic Valve Production

S. DEB & S. K. SEN

*Wireless Laboratory, University College of Science, Calcutta*

## Introduction

**T**HERMIONIC valves have now wide and varied uses in many fields such as radio and line communication, various forms of industrial control, electrotherapy, television, radar etc. This has led to the development of many varieties of valves from the simple diodes and triodes to tetrodes, pentodes, hexodes, octodes and their combinations. They vary in size from a tiny acorn, barely an inch long, to the giant transmitting valves, 4' high. They may have high vacuum inside or may be filled with vapour as in thyratrons, tungars, etc. The end of this amazing development of new types of valves is, however, not yet in sight. New types of valves with new uses are still being produced. In recent years valves like klystron, magnetron and phasitron, involving entirely new principles for work on microwaves, have been developed. This has been possible only through intensive fundamental research on the one hand and of similar developmental research on the other.

In view of the great importance of radio valves, specially because of its indispensability in appliances used by the communication and defence services, the *Board of Scientific & Industrial Research* in 1942, sanctioned a scheme of research on the laboratory production of radio valves in India. Unfortunately, on account of war conditions, the import of the necessary plant was greatly delayed. In 1945 a pilot plant built by the *Callite Tungsten Corporation*, U.S.A., was made available, and it arrived at the *University College of Science, Calcutta* in 1946. It took some time to put the plant into working condition as a number of its components had been damaged in transit. Work was, however, commenced in September 1946 in spite of the many difficulties such as non-availability of raw materials, etc. The two authors of the present note have successively been in charge of the plant, and they have worked out successfully the more important techniques in the manufacture of thermionic valves. In what follows, a description of the

plant and also an account of the details of the various processes involved in the construction of a thermionic valve of type 80 full-wave rectifier is given in detail, and brief references to other processes which can be worked with the plant are also given in the Appendix.

## The Pilot Plant

The pilot plant consists of the following :

- (A) Main working table with all the accessories
- (B) Booster unit, compound vacuum pump
- (C) High frequency induction furnace, and
- (D) Macleod gauge

(A) *The Main Working Table*—The various components comprising the unit (FIG. 1) are described below :

(1) Double wheel glass cutter — It consists of a cutting disc of carborundum (for soft glass) and another of steel (for hard glass) and is operated by a 110 volt A.C. motor, having 3,400 r.p.m.

(2) Five-burner adjustable cross-fires with ball joints — This comprises the following accessories with a 1/10 h.p. driving motor with reduction gear and universal head socket: (a) flare chuck, adjustable for different sizes; (b) stem head for making tipless stem; (c) sealing head for different bulb sizes; (d) basing head for all types of bases; and (e) equipment for blowing air through the tubes for making exhaust holes.

When making stems, the glass piece and lead wires are held in position by means of clamps provided and the stem head is rotated between cross-fires by means of a 1/10 h.p. motor. When it is desired to make flares, seal-in or to connect the base, the stem head is removed and is replaced respectively by flare chuck, sealing or basing head, according to the operation desired.

(3) Ribbon burner for melting long glass tubes — This is not of much importance for valve construction, but is essential for making neon signs.

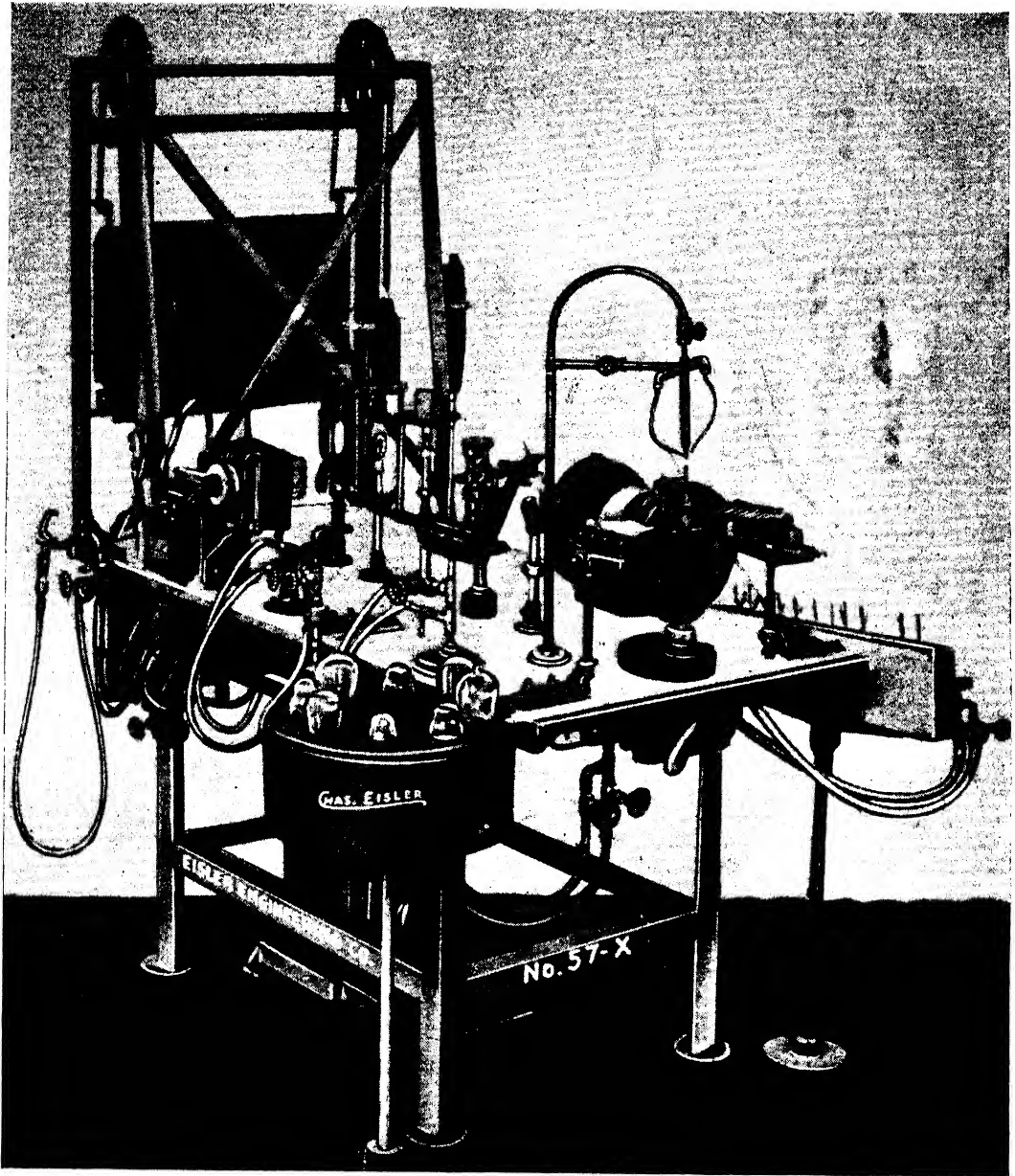


FIG. 1 — MAIN WORKING TABLE WITH ALL THE ACCESSORIES.

(4) Blast torch for glass fusion with arrangement for oxygen-air flame.

(5) Tipping torch for sealing off the valve.

(6) Needle flame burner for localized softening of glass at a point.

(7) Gas heated stem annealer — This consists of a long chamber inside which there are two parallel rows of flames. The distribution of the flames is such that the

temperature is maximum at one end of the chamber and diminishes towards the other end. The stem to be annealed is introduced at the hottest end and is taken out at the coolest end. The former is called the inlet and the latter the outlet end of the chamber.

(8) Gas-heated bulb rotary annealer — Useful for annealing of the glass envelopes after they have been sealed with stem.

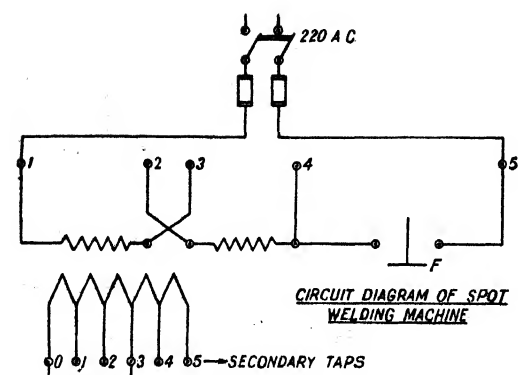


FIG. 2 — CIRCUIT DIAGRAM OF SPOT WELDING MACHINE.

(9) Three-head glass manifold with the gas-heated baking oven — The manifold is connected to the vacuum lines. The valves are sealed in the three exhaust heads and baked during the process of evacuation by the gas-heated oven. The oven can be raised or lowered by means of adjustable weights.

(10) Soldering iron with two burners — For soldering the lead-in-wires to the base socket.

(11) Spot welding machine — This consists of a single-phase power transformer with five secondary taps for varying the voltage in steps of one volt (FIG. 2). The particular voltage to be used depends on the thickness and the type of material to be welded. The welding is effected by the Joule heat generated between the metal surfaces to be welded when the current is passed in the primary of the transformer for a short time. The welder is operated by means of the foot-treadle (F) which closes the primary circuit when depressed. For finer control of welding voltage a rheostat may be included in the primary circuit. A proper weld is made by quick depression of the foot-treadle. For precision and quick working, the time of welding is to be controlled by an electronic circuit.

(12) Leak tester — This is a useful tool for locating leaks and for rough indication of pressure inside the vacuum system. It consists of a transformer, mounted in a cylindrical case provided with a small knob connected to a rheostat at one end and a metallic electrode at the other.

The tester is connected to the 110 volt A.C. line and the knob adjusted until the end of the electrode glows with a spark discharge. When held against the glass tubing of the pumping

system, the spark jumps to the glass and partially covers the outer wall of the glass. A poor vacuum is indicated by a dark-purple streak or red glow while a higher vacuum shows a light-blue colour which becomes thinner and clings close to the inside of the glass wall as the pressure decreases. The glow disappears entirely at pressures below  $10^{-4}$  mm. of mercury. A pale-red glow which gradually turns into a dark-purple streak inside the tube indicates a leak. The spark from the tester enters the tubing through the leak and becomes white in colour as it passes through the glass, the leak being indicated by a bright spot.

(B) *Vacuum Pump, Booster Unit, etc.* —

This is shown in Fig. 3. A motor (M) operated on 220 volts A.C. works both the compressor for the booster (for supplying gas-air mixture to the burners) and the pump unit through a common coupling shaft. The booster unit consists of 2 rotary compressors, one ( $C_1$ ) for air and the other ( $C_2$ ) for gas. The outlets of the compressors are connected to separate chambers of a reservoir drum (D). The pressure within the chambers can be controlled by adjustable mechanical lever arrangement (L) for gas and by an adjustable spring valve (S) for air. The unit supplies gas at 12" pressure on the water column — approximately equal to 7 oz. of pressure, and air at a pressure of 3 to 4 lb. — which are the conditions for best results. The compressed mixture of gas and air is circulated to various burners in the main working table.

The fore-pump (F) gives a vacuum of the order of  $10^{-4}$  mm. of Hg. This is followed by a mercury diffusion pump by means of which a pressure of the order  $10^{-6}$  mm. of Hg. can be obtained using a liquid oxygen freezing trap. The main vacuum line is a pyrex tube of wide bore to give the least possible resistance during pumping, and it is connected to a soft-glass manifold by a short connecting tube. Soft glass is used for the manifold to facilitate the sealing of the lead glass stem of the valve with the manifold heads.

(C) *High Frequency Induction Furnace* — The degassing of metallic parts is effected by heating in an induction furnace of spark oscillator type. The circuit diagram is shown in Fig. 4. The load to be heated is placed inside the coil (L) which is made of a hollow-copper tube. The coil together with the condenser and the load forms the tank circuit. The latter is coupled to the main spark generator circuit composed of

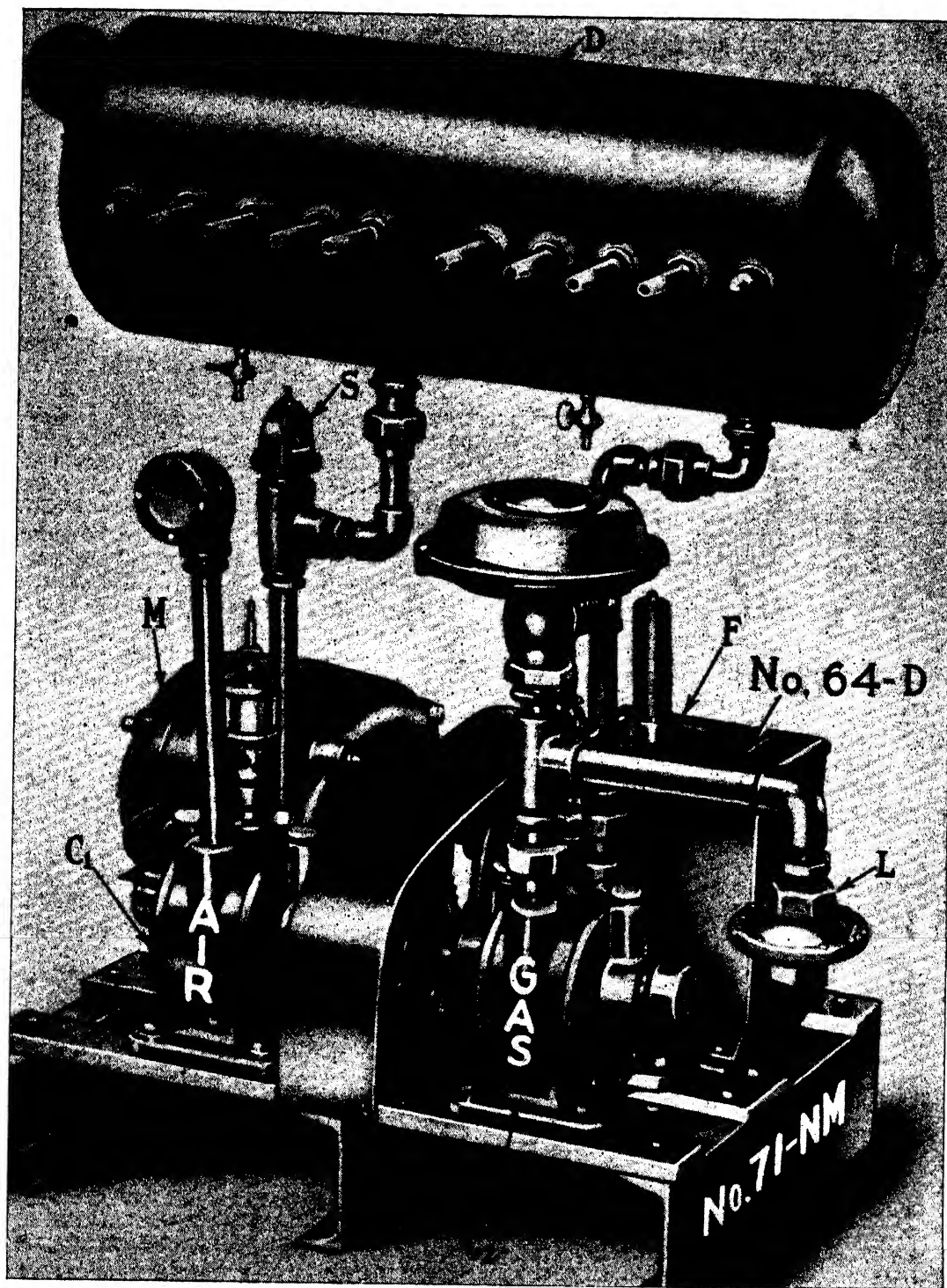


FIG. 3 — COMPRESSOR AND VACUUM PUMP UNIT — D: reservoir drum; M: motor; S: spring valve; F: fore-pump; L: mechanical lever; C<sub>1</sub>: compressor for air; C<sub>2</sub>: compressor for gas.

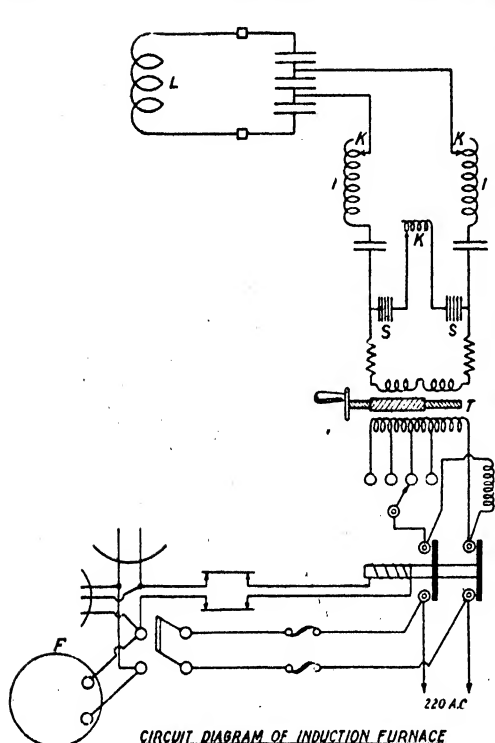


FIG. 4 — CIRCUIT DIAGRAM OF INDUCTION FURNACE — T: input transformer; SS: spark gaps; KK: tappings on the inductance coils; II: inductance coils; L: heating coil.

condenser, inductance and a set of tungsten spark gaps (SS). The tappings (K) on the coils can be adjusted for tuning. This adjustment is necessary because the heater coil has often to be changed in accordance

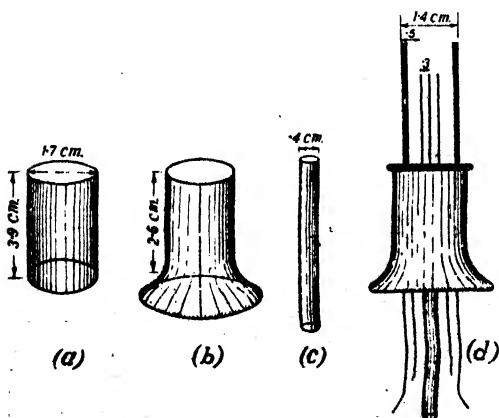


FIG. 5 — STRUCTURAL PARTS OF TYPE 80 FULL-WAVE RECTIFIER — (a) Glass piece for flare; (b) glass flare; (c) exhaust tube; (d) glass flare stem with lead-in-wires sealed.

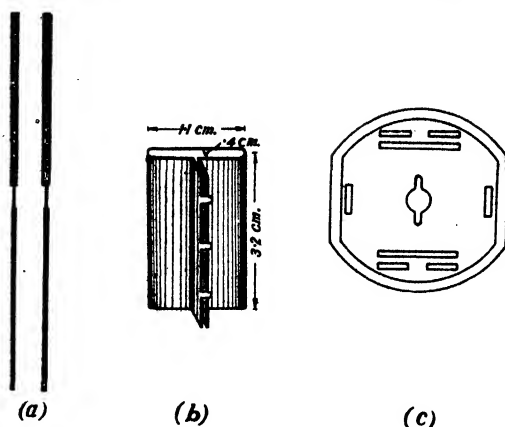


FIG. 6 — STRUCTURAL PARTS OF TYPE 80 FULL-WAVE RECTIFIER — (a) "Dumet" lead-in-wires; (b) anode plates of nickel; (c) mica spacer.

with the size of the load. Inductance (II) in the spark circuit may be varied within a range of 5 to 18  $\mu$ h. The input transformer (T) supplies power to the spark circuit. There is an adjustable magnetic shunt between the primary and the secondary coil of the transformer with the help of which the output of the furnace can be varied from 2.5 to 5.5 kVA. A thermocouple type of h.f. power metre coupled to the tank circuit serves as an indicator for the resonance condition of the two circuits. The width of the spark gap has to be adjusted to a value of 0.003" to 0.004" from time to time.

(D) *Macleod Gauge*— It is used for recording pressure up to  $5 \times 10^{-6}$  mm. of Hg. The mercury diffusion pump, using liquid oxygen in the freezing trap, gives a pressure of the order of  $10^{-6}$  mm. For accurate recording of higher vacuum, an ionization gauge is essential and is being constructed.

#### Construction of Type 80 Full-wave Rectifier

Type 80 is a directly heated, high vacuum, full-wave rectifier. It has got a maximum peak inverse voltage of 1,400 and gives a maximum D.C. output current of 125 m.a. Its filament consumes 2 amp. at 5 volts input. The constructional details of the valve are shown schematically in Figs. 5, 6, etc.

The various steps in the construction are the following:

- (A) Construction of the glass stem with lead-in-wires; annealing
- (B) Construction of electrodes
- (C) Cleaning and degreasing of materials; outgassing of metallic parts; carbonization of electrodes



- (D) Construction of filaments
- (E) Assembly of electrodes by spot welding; hooks and supports; mica spacers
- (F) Sealing of glass envelope
- (G) Evacuation and processes during evacuation, which include: (1) degassing, (2) cathode activation, and (3) gettering and sealing
- (H) Testing of finished valves
- (A) *Construction of Glass Stem with Lead-in-wires*—The different stages in making of the glass flare stem are shown in Fig. 5. A short glass piece (a) is first given the flare shape (b) by means of the flare head. The exhaust tube (c) and the lead-in-wires, fixed in the lead wire spacer, are then fused to the flare (d) by means of the stem head shown in Fig. 1. The exhaust hole at the terminal of the exhaust tube is then blown by means of the equipment shown in Fig. 1 by the combined effect of heating at a point and blowing air through the exhaust tube. The working temperatures for the different operations are shown in Table I.

TABLE I

OPERATION	WORKING TEMP., SOFT GLASS °C.	WORKING TEMP., HARD GLASS °C.
Flare	800	1,000
Stem	1,000	1,250

To prevent weathering due to the electrolysis effect resulting from the deposition of moisture on the bulb, the glass must possess a high electrical resistance, must be well annealed, must have a low working temperature and maximum lead-wire spacing. In our case, soft glass of the following composition was used, since it matches the thermal expansion while sealing with the lead-in-wires. Composition of soft glass:  $\text{SiO}_2$ , 57-72%;  $\text{Na}_2\text{O}$ , 5-8%;  $\text{K}_2\text{O}$ , 7-12%;  $\text{PbO}$ , 26-32%;  $\text{B}_2\text{O}_3$ , 0-2%;  $\text{Al}_2\text{O}_3$ , 0-2-0-8%;  $\text{Sb}_2\text{O}_3$ , 0-5%.

Lead-in-wires (Fig. 6a) are fused to the stem by the use of proper seals. In selecting the metal-in-glass seals the guiding principle is that when cooling after heating, the metal should contract at a slightly smaller rate than the surrounding glass and that the metal should have a good thermal conductivity. Obviously, this calls for different seals for soft glass (soda and lead glass) and hard glass (Pyrex and Nonex). For soft glass, "dumet" seals, which we use, satisfy the conditions of good thermal conductivity, quick dissipation of heat,

fairly cool operation and the required matching of thermal expansion with lead glass. "Dumet" seals consist of nickel-iron alloy with 48 per cent of nickel with a layer of copper which gives a better bondage with lead glass. Covering the wire with a layer of borax serves as a protection from excessive oxidation during sealing. "Dumet" seals, however, have complex expansion characteristics (radially  $10.25 \times 10^{-6}/0^\circ\text{C}$ . and longitudinally  $7.6 \times 10^{-6}/0^\circ\text{C}$ .). The effect of this can be minimized by the use of pieces of very short length. The uses of other types of metals in glass seal are given in Appendix A.

The different types of stems in use in modern valves are described in Appendix B.

Annealing is essential for removing any permanent strain set up within the material of the glass in cooling. If allowed to remain, it causes cracks when the finished product is put into service. The following two temperatures are important in connection with annealing.

*Annealing Point*—This is the temperature at which the viscosity is  $2.5 \times 10^{13}$  poises and 90 per cent of the strain is removed in 5 minutes from a glass sample of thickness  $\frac{1}{4}$ ". Physically, this represents the point at which there is sufficient mobility within the glass structure to permit the movement necessary to relieve any strains set up within a definite period of time (15 min.).

*Strain Point*—The strain point is the temperature at which the viscosity is  $4.0 \times 10^{14}$  poises and at which 90 per cent of the strain is removed in 4 hours in a glass sample  $\frac{1}{4}$ " in thickness. Physically, this represents the temperature below which no permanent strain is introduced at ordinary rates of cooling and above which annealed glass cannot be heated again without introducing permanent strain and requires re-annealing to free it from strain.

The finished work is first heated above the annealing point; the temperature is then gradually lowered following a schedule which depends on the hardness of the glass. The approximate schedule can be determined with the help of the equation given in Appendix D. In our case the approximate values of the annealing point and the strain point were  $430^\circ\text{C}$ . and  $390^\circ\text{C}$ . respectively. The stem immediately after construction is placed for a short while at the end of the annealing chamber where the temperature is slightly above  $430^\circ\text{C}$ . and annealed for about 5-10 minutes placing it at gradually



lower temperatures in the annealing chamber. The average temperature of the chamber is  $400^{\circ}\text{C}$ . The best annealing temperature and time were determined by repeated trials. It was found that it is best to cool comparatively slowly since the glass may crack, even though no permanent strain is introduced. A well-annealed glass has 2.5 to 3.0 times the electrical resistance of an unannealed glass.

(B) *Construction of Electrodes* — Anodes of the shape and dimensions shown in Fig. 6b were made from sheet nickel with the help of an ordinary dice and press machine. The choice of nickel for such valves is determined by the following considerations.

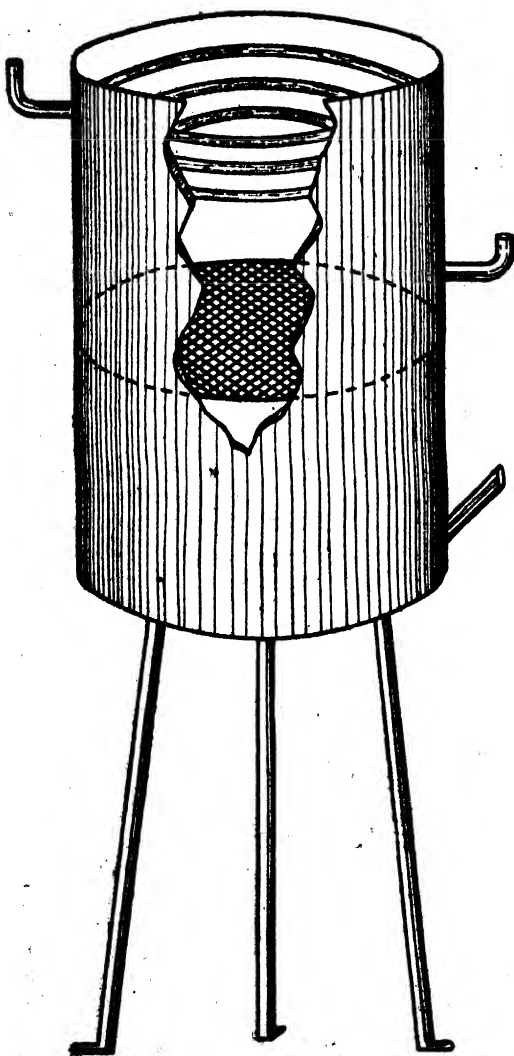


FIG. 7 — TRICHLORO-ETHYLENE DEGREASING CHAMBER.

High ductility, permitting construction of anodes of complicated shapes with less difficulty; freedom from transformation of the crystalline structure resulting in brittleness; low vapour pressure permitting high frequency heating in vacuum at  $100^{\circ}\text{C}$ .; low solubility of carbon permitting carbonization to increase heat dissipation; good strength and stiffness. Nickel, free from sulphur, is to be used since sulphur causes the grains of nickel to be separated by films of brittle sulphide.

It is to be noted that nickel is now generally replaced by nickel-plated steel although it is difficult to obtain an evenly distributed heavy plate of nickel on strip steel.

Iron and steel are used for cheapness but have the disadvantage of forming carbides during carbonization.

(C) *Cleansing & Degreasing of Materials: Outgassing of Metallic Parts & Carbonization of Electrodes* — It is essential that the glass tubes and the anode material be free from dirt, specially grease or oil. The slightest amount of grease or oil absorbed on the surface layer of the electrodes seriously hinders the attainment of high vacuum. Apart from this, subsequent heat treatment may burn the oil on the metal and spoil its surface or even affect its composition.

The glass tubes before use for flare or stem making or for bulbs for sealing into the stem, are, therefore, washed thoroughly with chromic acid, then with ordinary water and finally with distilled water and dried. Trichloro-ethylene, one of the best grease solvents known, was used for degreasing metallic electrodes. It has a low boiling point ( $87^{\circ}\text{C}$ .), low specific heat, low latent heat and has the decided advantage that it has no action on common metals. Degreasing by this chemical leaves the metal in a dry, neutral condition ready for succeeding treatment.

There are many types of degreasing plants used in industry. For our purpose, a simple device was designed and constructed in the laboratory (FIG. 7). It consists essentially of a cylindrical chamber divided into two compartments by wire gauge. In the lower chamber, a small quantity of trichloro-ethylene is boiled by means of a gas flame. Oily metallic parts are placed on the wire gauge. Cold water is circulated through pipes at the top which condense the trichloro-ethylene vapour from the degreasing chamber. The metallic parts are thus exposed to a vapour bath which dissolves the oil and falls into the sink. This treatment is

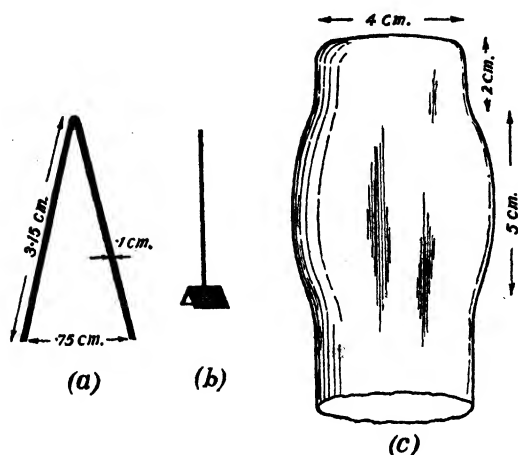


FIG. 8 — STRUCTURAL PARTS OF TYPE 80 FULL-WAVE RECTIFIER — (a) Oxide-coated filament; (b) getter tab of nickel; (c) glass envelope.

continued for half an hour by which time all traces of grease or oil are washed off. A thermometer can be introduced by the side hole for measuring the temperature.

It is also necessary to outgas the metallic parts before assembly. Unfortunately we were not able to do this for want of a proper vacuum furnace (this is under construction). The method of outgassing metallic parts before assembly is described in Appendix C.

The anode is given a coating of carbon. A carbonized anode keeps a lower temperature on account of efficient radiation. Heat dissipation is then increased and permits the use of a smaller anode area for the same dissipation thereby reducing the cost. Coating is given by spraying "aqua dag" (colloidal graphite) on the anode surface by a spraying gun. For securing a good deposit the surface must be properly cleaned.

(D) *Construction of Filaments*—The dimensions and design of the filament as used in the full-wave rectifier, type 80, is like those of standard type 80 valve. The filament is of the oxide-coated type (FIG. 8a). It consists of a strip of nickel of width 0.1 cm. to which is added 2 per cent aluminium. The strip is covered with a mixture of barium carbonate (approx. 40 per cent) and strontium carbonate (approx. 60 per cent). A length, 6.3 cm., of this strip is folded into V-shape. Carbonate coating from a length of about 2.5 mm. is removed from each end in order to facilitate spot welding of the filament with the supports. A pair of such V-shaped filaments is used in each of the valves.

(E) *Assembly & Spot Welding*—Great care is taken to avoid contamination by hand in all welding processes. For perfect welding, accurate adjustment and control of welding secondary voltages and the duration of welding is necessary. A blank trial with the metals to be used was carried out to determine the proper welding voltage and welding time. In the actual manufacturing process the welding time is automatically controlled by the use of a electronic circuit. Wires with different melting temperatures and heat conductivities weld together best when their diameters are related as follows:

$$\frac{d_1}{d_2} = \frac{K_2}{K_1} \sqrt{\frac{T_2 m_1}{T_1 m_2}}$$

where  $d$  is the diameter,  $Tm$  is the melting temperature and  $K$  is the heat conductivity.

This shows that for wires of the same material, welding will be more satisfactory when the diameters of the welded elements are equal.

This is found to be so in actual practice. The following Table II will serve as a rough illustration of the fact.

TABLE II		
ELEMENTS	BEST WELDING VOLTAGE	TIME OF CONTACT
0.5 × 0.5 mm. nickel wire	Step No. 3	Normal*
0.75 × 0.75 mm. nickel wire	Step No. 4	Normal
0.5 × 0.75 mm nickel wire	Step No. 4	Instantaneous (with wire of small diameter below)

\* The time of contact is neither too long, nor too short — 0.5 to 1.0 sec.

The anode and cathode supports are first welded in position according to design specifications. Mica insulators (FIG. 6c) cut according to the design are then fixed to the electrode for maintaining their relative spacings. The filaments are then shaped and welded to the support. Utmost care has to be taken so that the parts are not displaced or contaminated with dust or other external impurities during spot welding.

Nickel getter tabs to be used are first degassed by heating in vacuum and then a short piece of magnesium ribbon is fixed on it. In order to avoid any deposit forming on the electrode bounding system and thus short circuiting different electrodes, the getter deposit is directed towards the bottom of the bulbs by a suitable design and placing of the getter tab (FIG. 8b). To avoid ionization by electron bombardment of the gaseous molecules, which invariably remain in the residual getter, the getter is fixed to the filament supports to have the cathode potential.

**Support & Hooks** — Support and hooks should have sufficient ductility, thermal conductivity, high modulus of elasticity, stiffness and strength. Nickel has all these properties and is used as support wires.

**Mica Spacers** — Muscovite mica has good insulating properties and chemical stability up to a temperature of 500°C. and has been used by us. Above this temperature, however, muscovite mica rapidly decomposes with the liberation of water vapour — one of the most harmful gases in the valves. Phlogopite mica can be used up to a temperature of 800°C. For valves which operate at still higher temperatures, alumina, magnesia or steatite pressed from powdered material to the required shape and sintered at 1,500°-1,800°C. is used. Since mica contains 18 per cent combined water, it was baked at 200°C. before use.

(F) **Sealing of Glass Envelope** — Soda-lime glass of the composition:  $\text{SiO}_2$ , 69-73%;  $\text{Na}_2\text{O}$ , 13-16%;  $\text{K}_2\text{O}$ , 0.7%;  $\text{CuO}$ , 6.1-13%;  $\text{MgO}$ , 3.2%;  $\text{B}_2\text{O}_3$ , 1%;  $\text{Al}_2\text{O}_3$ , 1.3-5%;  $\text{Sb}_2\text{O}_3$ , 1% is used for glass bulbs (FIG. 8c) because of its cheapness and because it makes a good seal with the lead glass stem. Glass envelopes were supplied by *Bengal Electrical Lamp Works Ltd.*, Calcutta, and blown in our laboratory according to design and specification. Bulbs must be properly cleaned to avoid any stain formation for the following reasons:

(a) Glass heated to 400°C. evolves a small amount of hydrochloric acid which reacts in vacuum with the carbonate or the oxide of the cathode to give barium or strontium chloride.

(b) These chlorides, subsequently being heated, evaporate and condense on the grid and the anode and form stains.

(c) Under electron bombardment, the stain decomposes producing chlorine atoms or chlorine ions which poison the cathode emission to a pronounced degree. All traces of hydrochloric acid should, therefore, be removed before sealing.

The following cleaning schedule was found satisfactory:

The glass envelope is washed with chromic acid, then with distilled water and finally rinsed with caustic potash solution. It is then washed with distilled water, dried and sealed by the sealing head referred to in Fig. 1. The working temperature during sealing-in varies from 1,050°C. to 1,300°C. During the sealing-in of the stem with the glass envelope, care is taken not to dislocate

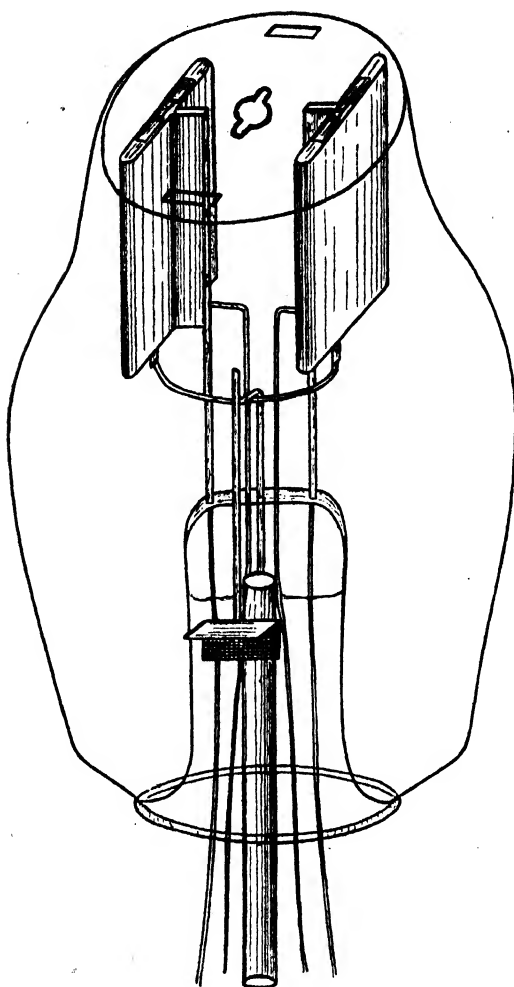


FIG. 9 — A TYPE 80 VALVE WITH ALL PARTS ASSEMBLED BEFORE EVACUATION.

any part. After sealing, it is annealed for 10-15 min. in the bulb-annealing chamber (annealing temperature nearly 500°C.). The complete structure of the valve before being evacuated is shown in Fig. 9.

(G) **Evacuation & Other Processes During Evacuation** — Rubber tubing connections are avoided as far as practicable in the vacuum line as they are often leaky and give out sulphur vapour in high vacuum which is harmful to radio tubes.

Water vapour to some extent is removed by using silica gel in a reservoir in the pumping line.

Leaks in the whole system are traced from time to time by the help of the spark coil described before.

The slightest trace of grease creeping in the pumping line is sufficient to destroy the vacuum. Use of grease in the stop cocks is avoided as far as possible and "apeizon" grease is used for high vacuum pump line. A valve has to be subjected to the following processes during evacuation :

(1) *Degassing* — There is always the danger that after setting up there remains some contamination of the surface. On glass walls, this is mainly water vapour. On metallic parts it is usually oil and grease. All parts are, therefore, carefully cleansed before assembly. In order to attain the highest vacuum it is necessary to heat the radio tubes on the pump.

First, the system is baked for half an hour at nearly 450°C. in a gas-heated oven to remove the vapour and gases adsorbed on the glass envelope and electrode parts at  $10^{-6}$  mm. pressure. For lead glass, the temperature should be 400°C., and for pyrex glass, 600°-650°C.

The nickel plates are then heated by induction heating coil to bright red heat 3 or 4 times for 10-15 sec. at intervals of 2 min. to remove the last trace of gas and heating is stopped only when there is no bluish-violet discharge of nickel vapour due to high frequency heating. The gas extracted from nickel is mainly carbon monoxide, carbon dioxide, hydrogen, small quantities of nitrogen and water vapour.

An idea of radio frequency power  $N_T$  necessary to raise a metallic electrode to a specified temperature  $T^\circ$  (abs.) may be obtained from the following formulae :

$$N_T = 5.75 \times 10^{-12} T^4 a \text{ watts}$$

$$N_T = 2.80 \times 10^{-4} A \sqrt{f \mu \rho_i} (n I)^2 \text{ watts}$$

where  $a$  is surface area of the metallic electrode;  $A$ , constant depending on the height of the cylinder to be heated; its diameter; and diameter of the eddy current heater coil (the thickness of the coil is negligible if below 0.3 mm.);  $f$ , frequency in cycles/sec.;  $\mu$ , magnetic permeability;  $\rho_i$ , specific resistance of the material heated in ohms/cm.;  $n$ , number of turns in the coil; and  $I$ , r.m.s. current in the coil.

Precaution was taken to see that (a) the temperature of heating was not above the point where the vapour pressure of the metal is greater than  $10^{-7}$  mm. of Hg. as otherwise volatilization of the metal will begin; (b) the pumping speed was made sufficient to remove as quickly as possible the gas evolved during heating as otherwise the gas evolved may

be ionized, giving rise to a discharge under the excitation of high frequency heating.

(2) *Cathode Activation* — Activation of filament means the proper formation of the surface of the cathode to give the required emission. In the case of oxide cathode, we use, barium-strontium coating is first decomposed by flashing the cathode. This is accomplished by heating the filament for a short time by a suitable filament power supply, which gives about 50 per cent input power above the normal. The carbon dioxide evolved is pumped out. The flashing time is critical and varies from 10 to 90 sec. The filament is immediately heated by 5 volts A.C., which is the normal filament operating voltage. Care is taken to see that the normal operating conditions are maintained from this time onward, because the filament is now in the active stage and any adsorption of gas due to cooling might spoil the life and activity of the filament. The critical time of flashing for the decomposition of carbonate coating for perfect emission is still under investigation.

During the decomposition of the carbonate coating, the eddy current heating of the anode and electrodes is resumed while the filament is still hot to remove any surface contamination due to carbon dioxide and free barium liberated from the filament during evacuation. The normal anode voltage is then applied. The anode current slowly rises and attains the value appropriate to the applied anode voltage in about 15 min. The building up of current is attributed to the gradual formation of an active layer of barium atoms on the surface of the filament through an electrolytic process.

(3) *Gettering Technique* — An appreciable amount of residual gas remains even after the above operations. There also remains some absorbed gases in the valve elements. These are liberated during the subsequent stage of operation, and unless these are removed, the valve will lose the vacuum. Chemically active substances called "getters" are used to absorb the residual gases. By the use of proper types of getters a vacuum of the order of  $10^{-7}$  to  $10^{-8}$  mm. can be easily maintained.

Gettering is carried out by any of the following processes :

(a) *Gettering by dispersion*, in which the vaporized metal combines rapidly with the residual gases in volume and the surplus of vapour settles on the cooler section of the bulb electrode.

FIG. 10 — AN ACTUAL PHOTOGRAPH OF STRUCTURAL PARTS OF TYPE 80 CONSTRUCTED — (a) Glass flare; (b) exhaust tube; (c) "dumet" lead-in-wires; (d) glass-flare stem with lead-in-wires sealed; (e) mica spacer — getter tab of nickel — nickel anode plates — "V" shaped oxide-coated filaments; (f) a glass flare stem with all parts assembled ready for sealing in glass envelope; (g) glass envelope; (h) valve ready for evacuation; (i) a finished valve with barium gettering.

(b) Gettering by contact, in which the getter deposited on the bulb surface combines slowly with the gases which come into contact with it due to temperature agitation.

(c) Gettering by ionization, during which a rapid chemical action takes place between the getter deposits and ionized gases.

No other getter being available locally or from foreign countries, we are now using magnesium getter which is fairly satisfactory. It getters by ionization. Contact action is slow, being limited by the coating of oxide layer formed. The getter is flashed at a

temperature of 500°-600°C. It should be noted that magnesium getter loses its activity if the tube is left unused for a long time.

When the pressure is of the order of  $10^{-6}$  mm. after the oxide formation by decomposition, the getter is flashed at a temperature of 500°-600°C. by restricting the eddy current heating to the region of getter tabs for 10-20 sec. and avoiding any undue heating of the other electrodes. Eddy current heating is stopped as soon as the getter is flashed and a good deposit is obtained.

Immediately after gettering, the tube is sealed off the pump line. It is found that a thick sealing maintains the vacuum well. A short account of the more effective getters which are actually used in the manufacture of radio tubes is given in Appendix E. An actual photograph of the structural parts leading to a finished type 80 valve is shown in Fig. 10.

(H) *Testing of Finished Valves* — (a) Emission tests: After sealing the tube off the pump, the valve is subjected to an activation schedule to complete the formation of the oxide-coated cathode. In general, a current of 100 ma. for 5-6 min. is sufficient for complete activation. A graph showing the current-voltage characteristic is prepared (Fig. 11). The graph also indicates the comparison with the characteristics of Philco type 80.

(b) Measurement of voltage regulation, peak diode current, diode impedances and

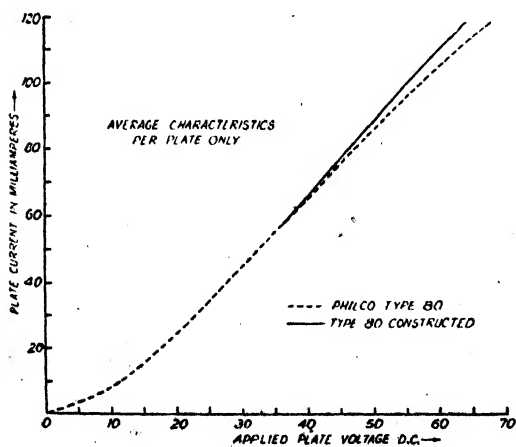


FIG. 11 — EMISSION CHARACTERISTICS OF TYPE 80 CONSTRUCTED COMPARED TO THAT OF Philco TYPE 80.

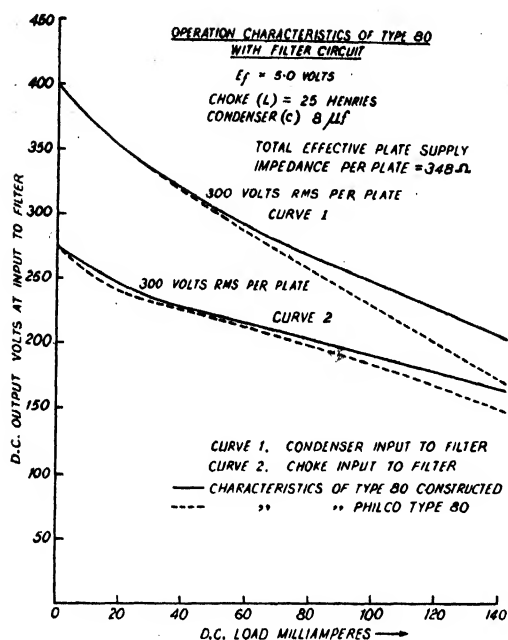


FIG. 12 — OPERATION CHARACTERISTICS OF TYPE 80 CONSTRUCTED WITH FILTER CIRCUIT COMPARED TO THAT OF *Philco* TYPE 80.

plate dissipation: Studies were made with 2 standard types of filter circuits, viz. (1) condenser-input circuit, and (2) choke-input circuit.

A typical set of curves for an input A.C. voltage 300 volts r.m.s. per plate is shown in Fig. 12. The corresponding curves for a *Philco* type 80 valve have been given for comparison. These curves provide a ready

method for the measurement of voltage regulation ( $\rho$ ), peak diode emission ( $\hat{I}$ ), peak diode resistance ( $\hat{r}_d$ ), average diode resistance ( $\bar{r}_d$ ), r.m.s. diode resistance ( $I_{rd} I$ ) and total plate dissipation ( $W$ ). Calculations were made for the following value:

Max. peak voltage,  $\bar{E} = 300 \sqrt{2}$  volts

External resistance,  $R = 348$  ohms

Capacity,  $C = 8 \mu\text{f}$

Choke inductance,  $L = 25$  Henries

For condenser-input circuit:

Type 80 constructed

(i)  $\bar{E} = 310$  volts

$\bar{I}_p = 25$  ma.

(ii)  $\bar{E} = 247.5$  volts

$\bar{I}_p = 50$  ma.

*Philco* type 80

(i)  $\bar{E} = 300$  volts

$\bar{I}_p = 25$  ma.

(ii)  $\bar{E} = 230$  volts

$\bar{I}_p = 50$  ma.

For choke-input circuit:

(i)  $\bar{E} = 225$  volts

$\bar{I}_p = 25$  ma.

(ii)  $\bar{E} = 192.5$  volts

$\bar{I}_p = 50$  ma.

(i)  $\bar{E} = 220$  volts

$\bar{I}_p = 25$  ma.

(ii)  $\bar{E} = 185$  volts

$\bar{I}_p = 50$  ma.

where  $\bar{E}$  is the D.C. output voltage and  $\bar{I}_p$  is the current per plate.

(c) Measurement of peak inverse voltage: A rough determination of the peak inverse voltage was made by gradually increasing the input A.C. voltage. Peak inverse voltage is given by the sum of the maximum peak input voltage that the tube can withstand and the corresponding D.C. output voltage. The values are recorded in Table III. The method of calculation has been briefly outlined in Appendix F.

TABLE III

QUANTITY	CONDENSER-INPUT CIRCUIT		CHOKe-INPUT CIRCUIT	
	Constructed type 80	<i>Philco</i> type 80	Constructed type 80	<i>Philco</i> type 80
$\rho$	0.61	0.74	0.89	0.97
$\hat{I}_p$	{ (i) 0.165 amp. (ii) 0.255 "	{ (i) 0.155 amp. (ii) 0.24 "	{ (i) 0.0594 amp. (ii) 0.108 "	{ (i) 0.059 amp. (ii) 0.107 "
$\hat{r}_d$	{ (i) 318.6 $\Omega$ (ii) 328.6 $\Omega$	{ (i) 426.0 $\Omega$ (ii) 443.6 $\Omega$	{ (i) 612 $\Omega$ (ii) 457 $\Omega$	{ (i) 712 $\Omega$ (ii) 532 $\Omega$
$\bar{r}_d$	{ (i) 302.0 $\Omega$ (ii) 373.4 $\Omega$	{ (i) 484 $\Omega$ (ii) 504 $\Omega$	"	"
$I_{rd} I$	{ (i) 342.5 $\Omega$ (ii) 353.2 $\Omega$	{ (i) 458 $\Omega$ (ii) 477 $\Omega$	"	"
$W$	{ (i) 6.12 watts (ii) 3.51 "	{ (i) 6.38 watts (ii) 4.44 "	{ (i) 5.768 watts (ii) 7.785 "	{ (i) 5.80 watts (ii) 7.66 "
Peak inverse voltage	1,368 volts (with 620 r.m.s. per plate)	1,400	1,363	1,400

### Discussion of Results

The curve accords with the general characteristics that the voltage regulation is better for a choke-input filter circuit. Compared to *Philco* type 80, the valve constructed by us shows a definitely better voltage regulation in both modes of operation and hence has a lower value of the valve impedance. Possibly, the much better regulation obtained for higher values of output current may be partly due to the presence of a trace of residual gas still left. This, if correct, should result in a lower peak inverse voltage and a diminished life of the valve. Measurement of the former quantity, however, shows that residual gas, if present, has a very negligible effect. No systematic test for life has yet been undertaken by continuous running of the tube although it should be noted that measurements were made about 4 months after the cathode had been activated.

### Materials Used in Construction

It will be interesting to examine the list of various raw materials used in the construction of the valve (type 80) and to see how far these materials are available in India. The following are the chief materials employed:

(1) Soda glass bulb, (2) lead glass tube, (3) mica insulator (muscovite or ruby), (4) dumet seals, (5) copper wire, (6) nickel wire, (7) nickel sheets, (8) magnesium ribbon, (9) oxide coated filaments, (10) Kulgrid hook wire, (11) trichloro-ethylene, (12) liquid oxygen, and (13) "Aqua Dag" (colloidal graphite).

Items 1, 3 and 12 are indigenous products and are available locally. Items 5, 8, 11 and 13 are not produced in India but are readily available in the market. All the remaining items have to be imported from the U.S.A. or the U.K. These materials can, however, be produced in India. We propose to develop a suitable process for the production of oxide-coated filaments in our laboratory.

### Acknowledgements

It is a pleasure to acknowledge with thanks the helps we have received. We received from *Messrs Metropolitan Vickers Electrical Co. Ltd.*, U.K., nickel sheets, wires and some special types of getters, free of cost, through the kind intervention of Sir Arthur Fleming and Dr. Kendall and from *Messrs Imperial Chemical Industries (India) Ltd.*, Calcutta, free supplies of certain chemicals for degreas-

ing, and also certain data. *Messrs Bengal Electrical Lamp Works Ltd.* have also rendered help in many ways. The work was carried out under the guidance of Prof. S. K. Mitra, D.Sc., and we thank him for his advice and constant interest in the progress of the work.

## APPENDIX A

### Types of Seal in Use in Radio Tubes

There are two types of seals, viz. (1) metal-in-glass seal; and (2) glass-in-metal seal.

(1) *Metal-in-glass Seal* — For this class of seals the metal should contract at a slightly lower rate than the surrounding glass. It is made by using "dumet" lead-in-wires (nickel-iron alloy with 48 per cent nickel and a layer of copper which gives a better bondage) with soft glass.

The alloy obtained by substitution of cobalt for nickel in nickel-iron lead-in-wires has lower expansion than soft glass and can be used in hard glass seals.

Molybdenum is found suitable for metal-in-glass seal with hard glass ( $\text{SiO}_2$ , 73%;  $\text{Na}_2\text{O}$ , 2.4%;  $\text{K}_2\text{O}$ , 4.2%;  $\text{CaO}$ , 3%;  $\text{B}_2\text{O}_3$ , 4.6%;  $\text{Al}_2\text{O}_3$ , 3%).

(2) *Glass-in-metal Seal* — For this class of seals, the glass should contract at a lower rate than the metal after heating. Chromium-iron alloy with 27 per cent chromium has an expansion  $10.5-11 \times 10^{-6}/^\circ\text{C}$ . which is greater than soft glass and is suitable for soft glass in the metal type of seals.

Tungsten can also be used for this class of seals. Its low thermal expansion matches that of "Nonex" glass ( $\text{SiO}_2$ , 75%;  $\text{Na}_2\text{O}$ , 3.4%;  $\text{K}_2\text{O}$ , 1.9%;  $\text{CaO}$ , 0.3%;  $\text{B}_2\text{O}_3$ , 18.3%;  $\text{Al}_2\text{O}_3$ , 1%) and the adhesion of oxide to tungsten is great enough for making glass-metal seals even for the leads of power and transmitting tubes. To avoid excessive oxidation, the tungsten wire is beaded. Its high electrical conductivity is a decided advantage in seals which are to carry high frequency currents.

An alloy of cobalt-nickel-iron containing 18 per cent nickel, small amount of other impurities and molybdenum, called "Fenico", is also used in metal tubes.

## APPENDIX B

### Types of Stems in Use in Radio Tubes

There are 4 types of stems: (1) glass-flare stem; (2) metal tube stem; (3) metal tube button stem; and (4) all-glass button stem.

(1) The glass-flare stem consists of a glass tubing with one end formed into a flare and the opposite end pinched to form the seal containing the leads. The stem length for glass-flare stem in modern receiving valves ranges from 29 mm. to 14 mm. The total clearance usually allowed between the flare and the inside walls of the base ranges from 5 mm. to 2 mm. The clearance between the leads in flat-press stem should be increased to eliminate possible difficulty of electrolysis.

(2) In a metal tube stem, each lead is brought into the tube through an individual metal eyelet and made vacuum tight by means of a glass seal. This is not much used on account of the high cost.

(3) In a metal tube button stem, a glass button is moulded into a metal sleeve. The metal sleeve is welded to a cold, rolled-steel flange. The whole assembly is then loaded onto a mould in which the lead wires and tubing are located, with one short length of glass tubing outside and another inside. The glass is then pressed into the metal sleeve by heating.

(4) All-glass button stem is similar to the metal tube button stem except that the metal sleeve is omitted. The use of this kind of stem reduces the over-all length of the tube slightly and improves shielding.

## APPENDIX C

### Outgassing of Metallic Parts

#### Before Assembly

Although degassing is done by the induction furnace heating or by electron bombardment during evacuation, previous outgassing of the metallic parts is important in attaining a high order of vacuum and in maintaining the same during the operation of the valve. The common method of outgassing consists in heating the metallic parts in a suitable vacuum furnace under a pressure of  $10^{-4}$  mm. The metal electrode to be outgassed is placed in a molybdenum boat and inserted into the furnace. A temperature of  $750^{\circ}$ - $950^{\circ}$ C. is suitable for degassing nickel and its alloys.

Outgassing temperatures for some of the common materials are given below.

Material	Outgassing temperature, °C.
Tungsten	1,800
Molybdenum	950
Tantalum	1,400
Platinum	1,000
Copper and copper alloys	500
Graphite	1,500-1,800

Some metals, e.g. molybdenum, platinum, nickel and iron may be cleaned by heating in an atmosphere of hydrogen. The samples are heated at  $950^{\circ}$ C. in a silica tube furnace containing dry hydrogen at a pressure of  $10^{-3}$  mm. of Hg. Heating in a dry hydrogen atmosphere introduces hydrogen into the metal by adsorption but owing to its high rate of diffusion, it is entirely removed by baking at  $450^{\circ}$ C. and rendered hydrogen-free.

## APPENDIX D

### Annealing Temperature

The initial rate of cooling,  $h_o$ , in annealing may be expressed as follows:

$$h_o = \frac{BF}{C} = \frac{\Delta N_s}{C}$$

where  $h_o$  is expressed in  $^{\circ}$ C./min.;  $B$ , birefringence constant;  $F$ , stress in kg./cm.;  $\Delta N_s$ , final stress in optical unit = 2.5 millimicron/mm. of glass thickness;  $C$ , constant =

$$\frac{\alpha \beta a^2}{6k \left( \frac{1}{6R} + \frac{2}{9K} \right)}$$

$\alpha$ , coefficient of expansion;  $k$ , thermal diffusivity, =  $\frac{\text{heat conductivity}}{\text{density} \times \text{specific heat}}$ ;

$R$ , modulus of rigidity;  $K$ , modulus of compressibility;  $a$ , semi-thickness of glass.

After the initial cooling, the rate is  $h_x$ , where

$$h_x = \frac{h_o}{a} \left( 1 + 2 \frac{T_o - T_x}{20} \right)$$

where  $T_x$ , new temperature in  $0^{\circ}$ C.;  $T_o$ , original temp. in  $0^{\circ}$ C., and  $h_o$ , initial cooling rate.

Initial cooling rate in terms of thickness for pyrex glass

$$= \frac{\Delta N_s}{3.2a^2}$$

The whole of the glass should be maintained at a uniform temperature, higher than the annealing temperature, for a short time after which it is gradually cooled in the annealing chamber through about  $100^{\circ}$ C. at a rate not exceeding  $20/t^2$  degrees per minute (where  $t$  is the wall thickness in mm.) to take it below the strain point. A cooling rate of  $100/t^2$  degrees per minute should never be exceeded, the cooling being at as uniform a rate as possible. The rate of cooling afterwards can be rapid as no strain is introduced after



this stage. Table A gives the temperature ranges for some common types of glass.

TABLE A

TYPE OF GLASS	ANNEALING TEMP., °C.	STRAIN TEMP., °C.	SOFTENING TEMP., °C.
Lead glass	425-435	389-398	126-630
Hard glass	647-818	450-510	697-756
Soda-lime glass	510	475	696

For lead glass, used by us, we have the following values :

$$R = 0.0040 \text{ sq. cm./sec.}$$

$$\alpha = 87 \times 10 / 0^\circ\text{C.}$$

$$B = 3.2 \times 10 \text{ due to 1 kg./sq. cm. load}$$

$$K = 0.35 \times 10 \text{ kg./sq. cm.}$$

$$h = 0.24 \times 10 \text{ kg./sq. cm.}$$

C, as calculated according to the above formula, has the value  $14.85a^2$ , where  $a$  is the semi-thickness of the glass. Therefore, the cooling rate per minute is :

$$h_0 = \frac{\Delta N_s}{C} = \frac{2.5}{14.85 \times 0.0025} = 67.5^\circ\text{C./min.}$$

According to the above formula, we have the following values for the type of lead glass used by us ( TABLES B and C ).

TABLE B

Temperature interval =  $20^\circ\text{C.}$

GLASS THICKNESS 1 mm.		GLASS THICKNESS 1.5 mm.		GLASS THICKNESS 2.0 mm.	
Cooling rate, °C.	Time interval, sec.	Cooling rate, °C.	Time interval, sec.	Cooling rate, °C.	Time interval, min. sec.
$h_0 = 67.5$	17.4	30	39.6	16.8	1 12
$h_{20} = 101.25$	11.54	45	26.6	25.2	47.6
$h_{40} = 168.75$	7.20	75	16.0	42.0	28.5
$h_{60} = 303.75$	3.70	135	8.8	75.6	15.8
$h_{80} = 573.75$	2.04	255	4.7	142.8	8.4
$h_{100} = 113.75$	1.02	495	2.4	277.2	4.3

TABLE C

GLASS THICKNESS 2.5 mm.		GLASS THICKNESS 5 mm.	
Cooling rate, °C.	Time interval, min. sec.	Cooling rate, °C.	Time interval, min. sec.
$h_0 = 10.8$	1 48	2.7	7 24
$h_{20} = 16.2$	1 14.4	4.05	4 54
$h_{40} = 27$	44	6.75	2 54
$h_{60} = 48.6$	24.4	12.15	1 36
$h_{80} = 91.8$	13	22.95	52.2
$h_{100} = 178.2$	6.7	44.55	26.9

## APPENDIX E

### Types of Getters in Use in Radio Valves

The following are the types of getters in common use :

(a) *Copper-nickel-iron or Aluminium-clad Barium Getters*. — A stick of barium is worked into a wire by rolling or drawing. A 0.25" length of this assembly is taken and used as getter. Vaporization of the getter occurs

at  $1,000^\circ\text{C.}$  when the internal pressure is great enough to eject barium vapour together with the molten metal and molten barium from the sheath. Sheaths of nickel, aluminium or iron may be used with equal advantage. This kind of getter is widely used in large glass receiving tubes. Barium gives instead of a silvery, a dark deposit when it absorbs gas. For metal tubes, iron wire having a barium core is taken and a section of this is used as getter. Barium from the interior bursts out through the inner walls permitting the getter to deposit to the section of bulbs in a plane roughly parallel with the thin edge.

(b) *Barium Azide Getter* — A nickel strip with a central hole is dipped into a concentrated solution of barium nitrite in water and then allowed to dry. During the bake out, nitrogen is released at  $150^\circ\text{C.}$  and the remaining barium acts as a getter. This getter gives a dark deposit. Barium azide getter is not generally used for it is explosive and azide solution is poisonous.

(c) *Barium-magnesium & Barium-aluminium Alloy* — An alloy of barium may be

used as getter owing to the large difference in vapour pressure of barium and magnesium. A large amount of magnesium gets evaporated before barium disperses. The alloy is stable in air if the barium content is 25 per cent. A serious disadvantage of this getter is that gettering by barium cannot be visually observed.

A barium-aluminium alloy with 50 per cent barium can be used as getter with nickel or iron tubes. No aluminium is dispersed before barium and visual observation of gettering is possible. Barium-aluminium alloy has a flashing temperature of  $700^\circ\text{C.}$  to  $1,700^\circ\text{C.}$  permitting the use of nickel-iron getter tabs. Magnesium may be mechanically mixed with barium-aluminium alloy or a mixture of barium-magnesium and barium-aluminium alloy may be used, for it is advantageous to use magnesium as pre-cleaning agent.

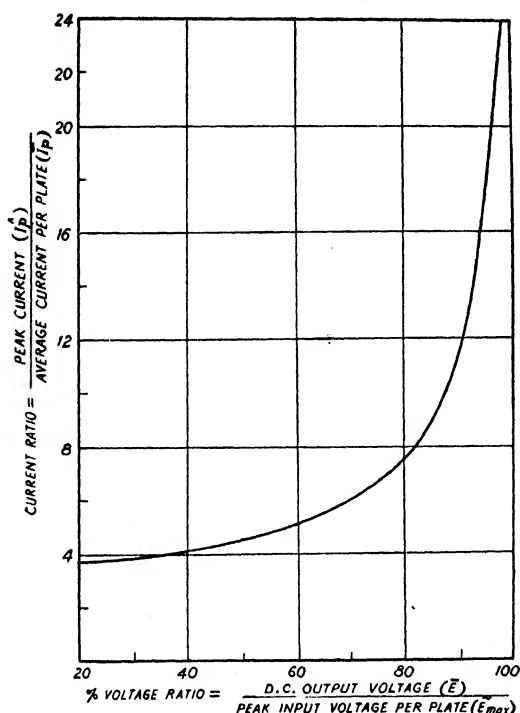
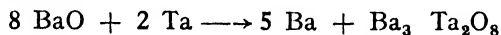


FIG. 13 — CURRENT RATIO AND VOLTAGE RATIO CHARACTERISTICS OF FULL-WAVE RECTIFIER.

(d) *Alkali Metals* — Because of their high vapour pressure, these metals are not suitable as getters in receiving tubes but are used in special tubes like caesium-vapour detector tube, photo tube, vapour lamps and gas discharge devices.

(e) *Batalum & Batalum Ribbon Getters* — These are used in metal tubes. Tantalum wire is coated with barium and strontium carbonates which are converted into oxides at 1,000°-1,100°C. and finally to metal vapour at 1,300°C. The reaction is as follows:



(Temperature of reaction is 1,300°-1,500°C.) Batalum ribbon getters consist of a tantalum strip with the middle portion formed into a channel. A barium compound with nitrocellulose binder is filled into the channel on the concave side. The use of barium-baryllite removes the difficulty of decomposition of carbonate during exhaust.

(f) *Misch Metal* — It is a mixture of rare earth metals and contains much cerium and lanthanum and, in addition, a variable amount of sodium not exceeding 5 per cent. Misch metal is inferior to barium, since it

becomes covered with a monomolecular film of oxide. It is now used in gas-filled regulators as an oxygen getter and as a means to maintain uniformity and magnitude of cathode fall during the life.

## APPENDIX F

### Method of Calculation of Voltage Regulation, Diode Impedances, Plate Dissipation, etc., of Type 80 Full-wave Rectifier

By voltage regulation characteristic of a rectifier circuit is meant the relationship between the output voltage and output current. Voltage regulation  $\rho$  is defined as

$$\frac{E_{\text{no load}} - E_{\text{full load}}}{E_{\text{full load}}} = \rho \quad (1)$$

It should be noted that  $\rho$  depends on the external resistance induced in the circuit and the type of filter circuit. Studies were made for two standard type of filter circuits:

- (1) Condenser-input circuit (FIG. 14A), and
- (2) choke-input circuit (FIG. 14B).

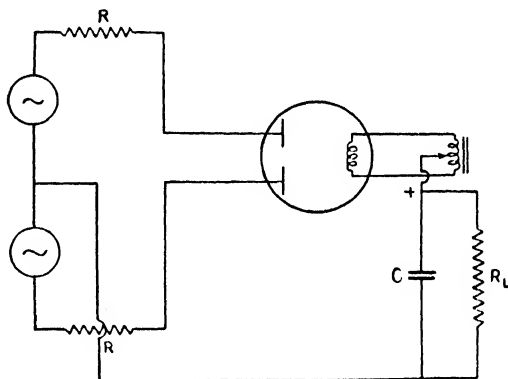


FIG. 14A — CONDENSER-INPUT RECTIFIER CIRCUIT.

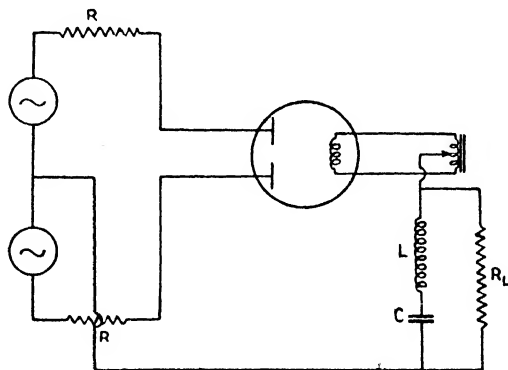


FIG. 14B — CHOKE-INPUT RECTIFIER CIRCUIT.

For measurement of  $\rho$ ,  $E_{full\ load}$  was taken at the output current of 100 ma. in eqn. (1).

For the measurement of peak diode current and valve impedances, the cases of the two filter circuits shall have to be treated differently.

Fig. 14A shows the diagram of a condenser-input filter circuit.  $R$  represents the stray resistance induced in the circuit due to the input transformer. In this case plate current flows in short pulses during the period for which the A.C. applied voltage is positive at the plate in question and in excess of the D.C. output voltage. The value of equipment diode resistance, therefore, depends on whether we are analysing for peak diode current, average diode current, etc. Thus, peak diode resistance  $\hat{r}_d$  is given by

$$\hat{r}_d = \frac{\hat{e}_d}{\hat{I}_p}$$

where  $\hat{e}_d$  is the peak diode voltage and  $\hat{I}_p$  is the peak diode current.

The average diode resistance  $\bar{r}_d$  is defined as  $\bar{r}_d = \frac{\bar{e}_d}{\bar{I}}$  where  $\bar{e}_d$  and  $\bar{I}$  are the average diode voltage and current during the conduction period.

R.m.s. diode resistance  $I r_d I$  is defined as the resistance in which the power loss due to a current having the same value of r.m.s. diode current is equal to the plate dissipation  $W_p$  of the diode.

For all practical cases, the following relations hold within an accuracy of 5 per cent.

$$\hat{r}_d = 0.88 r_d = 0.93 | r_d |$$

To determine  $\hat{I}$  and  $\hat{r}_d$ , we proceed as follows:

It has been shown by Kauzmann that if the input condenser is assumed to be very large and the current pulses to be half sinusoidal, the following relation holds:

$$\frac{\hat{I}}{\bar{I}} = \frac{\bar{A}}{2} \frac{180^\circ}{\cos^{-1} \frac{\bar{E}}{\bar{E}}}$$

where  $\bar{E}$  is the D.C. output voltage and  $\bar{E}$  the peak inverse voltage.

The relation has been shown graphically in Fig. 13. From this curve  $\hat{I}$  can be deter-

mined if  $\bar{E}$ ,  $\bar{E}$  and  $\bar{I}$  are known. The value of  $\hat{r}_d$  can now be determined from the relation  $\hat{I} (R + \hat{r}_d) + \bar{E} = \bar{E}$ . Knowing  $\hat{r}_d$ ,  $\bar{r}_d$  and  $I r_d I$  can also be calculated.

Power dissipation per plate is given by

$$W_T = W_p + \frac{1}{2} E_f I_f$$

where  $W_p = 0.84 \hat{I} \hat{r}_d I$  and  $E_f I_f$  = filament input power.

Fig. 14B shows the diagram of a rectifier circuit using choke-input filter. Here again  $R$  represents the stray resistance due to the input transformer. It can be shown that provided

$$L \geq \frac{0.91 \bar{E}}{6\sqrt{2} \bar{A} f \bar{I}}$$

where  $L$  = choke inductance in Henries,  $f$  = the frequency of the input A.C. voltage, the diode current approximately consists of rectangular pulses (except for a small first harmonic ripple at the top) and flows over an entire half cycle of the applied voltage. For such a case it is clear that

$$\hat{r}_d = I r_d I = \bar{r}_d = r_d \text{ (say)}$$

In our case for example  $\bar{E} = 300 \sqrt{2}$

$$f = 50$$

$$\bar{I} = 0.1 \text{ amp.}$$

$$L \geq \frac{0.91 \times 300}{300 \bar{A} \times 0.1} \geq 3H$$

The value of inductance used was 25 H and hence the relation is applicable.

Output current for a full-wave, choke-input rectifier, however, contains an appreciable portion of 1st harmonic of frequency  $2f$ . It may be shown that the peak diode current

$$\hat{I} = \bar{I} + \frac{0.471 \bar{E} \sqrt{2}}{4 \bar{A} f L}$$

No load output voltage is approximately given by

$$\frac{0.91 \bar{E}}{\sqrt{2}}$$

Hence for an output voltage  $\bar{E}$  at load current  $\bar{I}$

$$\frac{0.91 \bar{E}}{\sqrt{2}} - \bar{E} = \bar{I} (r_d + R)$$

The total plate dissipation is given by

$$W_T = W_p + W_f$$

where

$$W_p = 0.5 \left( \bar{I}^2 + \frac{0.03 \bar{E}^2}{\bar{A}^4 f^2 L^2} \right) r_d$$

and  $W_f$  = filament input power.

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## Fretting Fatigue—Literature Report No. 1

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**F**RETTING fatigue, also called rubbing fatigue and chafing fatigue, is a particular type of fatigue failure, which is probably related to both notch fatigue and corrosion fatigue<sup>1,2</sup>. The main characteristic of these types of fatigue is a combination of two factors, (a) the repeated loads or vibrations which define the load applications as fatigue or endurance and (b) a factor which reduces the fatigue strength and endurance limit obtained in the absence of this damaging factor.

In the case of fretting fatigue, the damaging factor consists of a close contact between the part subject to vibration and another part which is either fitted on to the vibrating part or in gliding contact with it. The most important example of premature failure due to fretting fatigue is that of railway axles on to which the wheel has been press fitted on shrunk<sup>3-16</sup>. Fundamentally the same failure has been also observed on shafts<sup>4,11</sup>, and on crank-pins<sup>16,18</sup>. Various types of joints differ from these fitted members, in that none or only a slight pressure is present during rest conditions; however, the pressures resulting from the vibrations also cause fretting fatigue failures. The most important instance of such a joint is that between an aircraft propeller and its hub<sup>1,19,21,25,40</sup>. Other cases of this type are screws or bolts and nuts<sup>23,28</sup>, keyed joints<sup>26</sup>, couplings<sup>27</sup>, joints between wire or wire rope and clamps<sup>28,29</sup>, and attachments of springs<sup>30,31</sup>. Failures between gliding members are those of parts running in bearings<sup>32</sup> and of gear teeth<sup>32</sup>. Possible, the

fatigue failures of the bearings themselves are also due to fretting fatigue. However, these will not be considered here. A large variety of fretting fatigue failures have been described by Thum and Wunderlich<sup>33</sup>.

Most observations on fretting fatigue relate to mild steel. However, most metals and alloys appear to be susceptible to a damaging effect by fretting. It has been found to be present in aluminium alloys<sup>1,19,22</sup>, magnesium alloys<sup>19,22</sup>, copper alloys<sup>1,19,22</sup>, and stainless steels<sup>1</sup>.

The type of fatigue failures observed at contact surfaces are obviously the same for contacts either in relative rest or in relative movement. This is proved by the fact that the laws of fretting fatigue failures have been established by extensive laboratory tests<sup>1,19,27,28,33-42</sup> which are predominantly of the type in which failure occurs at the contact between the rotating specimen and the bearing.

The literature on fretting fatigue has been repeatedly abstracted and critically discussed<sup>2,43-46</sup>.

The fatigue failure, under conditions where fretting is present, occurs in all instances either at the edge of the contact area between the two contacting members or close to it but within the contact surface.

The failure may occur in either one or in both contacting members.

In many instances of fretting fatigue, the production of abrasive products has been observed<sup>40,47-51</sup>. This process is usually called "bleeding", the term being illustrative for the red colour of the abrasion

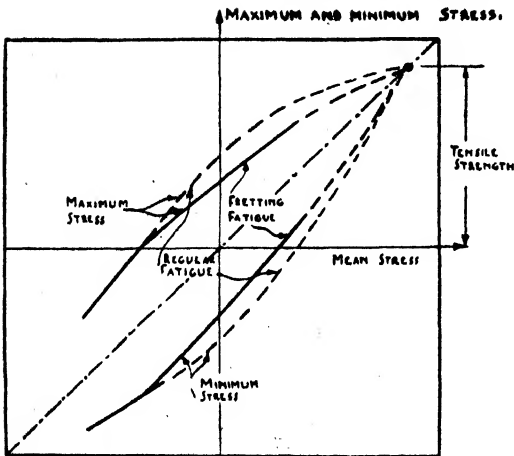


FIG. 1 — DIAGRAMMATIC REPRESENTATION OF THE RELATION BETWEEN FRETTING FATIGUE STRENGTH AND REGULAR FATIGUE STRENGTH FOR VARIOUS CONDITIONS OF AXIAL LOADING.

products of steel. The absence of such abrasion products, however, is no proof that fretting does not occur and that the fatigue strength is unaffected.

The laws of fretting fatigue, as far as they have been recognized, are generally the same as those for notch fatigue and corrosion fatigue<sup>1</sup>. The most important of these laws are the following:

(a) A damaging effect probably occurs only in the presence of applied tensions. On the contrary, it appears that fretting in the absence of tensions, i.e. in the presence of compressions only, would not adversely affect the fatigue strength. This is illustrated schematically in Fig. 1. However, definite information in this respect does not seem to exist at present. The graph in Fig. 1 illustrates that the fatigue strength is reduced less, the more the load condition changes from tensions only (upper right quadrant) to tension-compression, and that the damaging effect is nil in the range of compressions only (lower left quadrant).

(b) The damaging effect for a given load application such as rotating bending is highly dependent upon the material<sup>1</sup>. For certain groups of materials such as aluminium alloys, the fretting fatigue strength has been found, according to Fig. 2, to be equal to the regular fatigue strength (determined for polished specimens) if this value was very low. With increasing regular fatigue strength (or tensile strength, or hardness), the effect of fretting becomes increasingly larger. This results in the fact that for high strength alloys, the fatigue

strength in presence of the damaging effect (under conditions of either severe fretting, notching or corrosion) is practically independent of either the regular fatigue strength or the tensile strength.

The fretting fatigue strength is also dependent, according to Fig. 2, upon the structural condition of an alloy, to a slight extent. Cast alloys are apparently more resistant than annealed which, in turn, are more resistant than either cold-worked or heat-treated alloys, on the basis of equal strength.

(c) The damaging effect increases with increasing loads. As shown in Fig. 3, it becomes generally apparent only if the load range is sufficiently low to cause failure after a considerable number of cycles,

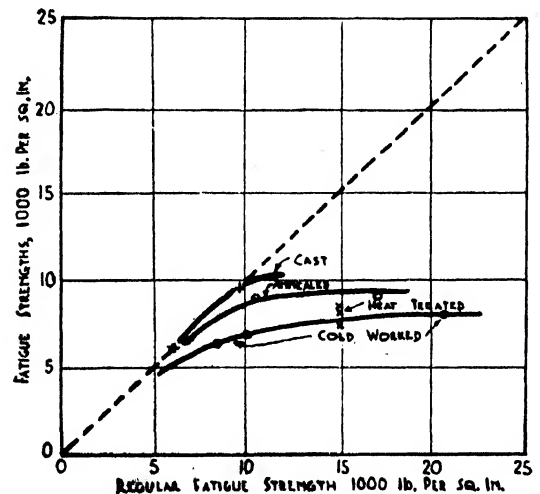


FIG. 2 — RELATION BETWEEN FRETTING FATIGUE STRENGTH AND REGULAR FATIGUE STRENGTH FOR VARIOUS ALUMINIUM ALLOYS (SACHS-STEFAN<sup>1</sup>).

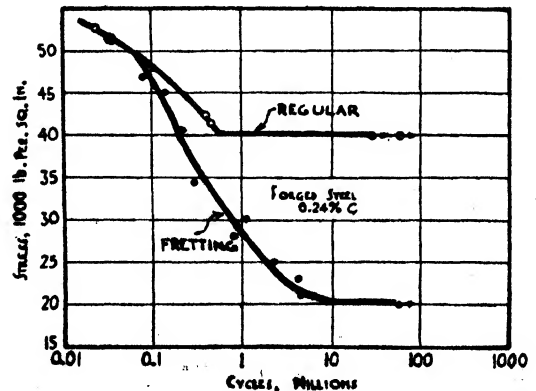


FIG. 3 — STRESS CYCLE CURVES FOR TESTS ON FRETTING FATIGUE AND REGULAR FATIGUE OF A FORGED STEEL (SACHS-STEFAN<sup>1</sup>).

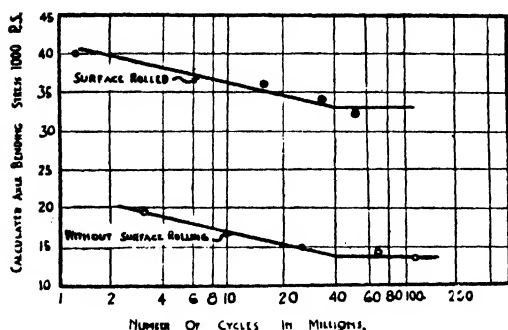


FIG. 4 — EFFECT OF SURFACE ROLLING ON THE FRETTING FATIGUE STRENGTH OF S.A.E. 1045 STEEL AXLES (BUCKWALTER-HORGER<sup>4</sup>).

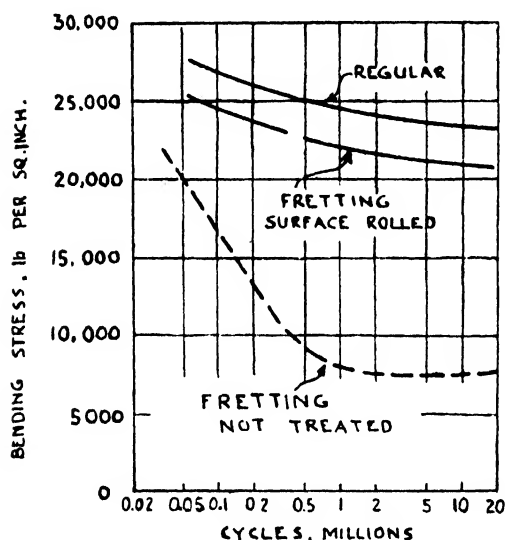


FIG. 5 — STRESS-CYCLE CURVES FOR REGULAR FATIGUE FOR FRETTING FATIGUE AND FOR FRETTING FATIGUE OF SURFACE ROLLED SPECIMENS OF A MAGNESIUM ALLOY (BOWDEN AND RIDLER<sup>50</sup>).

anywhere above 10,000 cycles at least. Furthermore, as shown in Fig. 3, stress-cycle curves indicate that no true fretting endurance limit exists, the value of the fatigue strength steadily decreasing with increasing number of cycles.

(d) Certain mechanical and thermal treatments eliminate the greatest portion of the damaging effects.

The mechanical treatments used to thus improve the life of parts, particularly of railroad axles, are surface rolling with one or several polished rolls,<sup>6,9,26,52-54</sup> and peening or shot-blasting<sup>9,55-57</sup>. The effects of such a treatment is illustrated in Figs. 4 and 5. The improvement resulting from such processes depends upon the processing

conditions such as the roll size and rolling pressure in surface rolling<sup>6,21</sup>, as shown in Fig. 6, or the shot-size and pressure in shot-blasting<sup>55</sup>.

By flame hardening the contact surface, an improvement similar to that obtained by mechanical treatments can be achieved<sup>7,9,57</sup>.

It appears that these beneficial effects are only attributable to a minor extent to a structural improvement of the material. The major portion of the improvement is explained by the introduction of compressive surface stresses by either the mechanical or the thermal treatments<sup>11,21</sup>, which prevent tensile stresses to develop during the vibrations. The relation between the magni-

KEY	SPEC. DIA.	ROLLER SIZE & FEED DIA.	CONV. R.P.D.	PER INCH	S.A.E. 1045 STEEL NORM. & DRAWN
—○—	0.3"	0.44"	0.03"	224	1/2 DIA. COLD ROLLED.
—△—	1"	1.46"	0.31"	60	1 1/2 " " " "
—□—	2"	2.92"	0.63"	60	3 1/2 " " " "

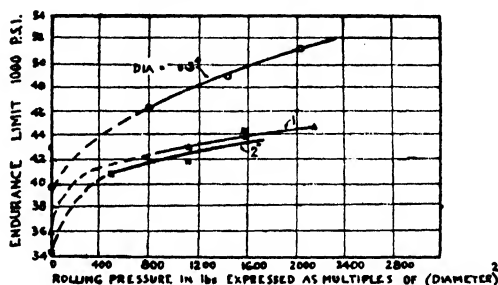


FIG. 6 — EFFECT OF ROLL PRESSURE ON THE FRETTING FATIGUE STRENGTH OF SURFACE ROLLED, S.A.E. 1045 STEEL AXLES OF VARIOUS SECTION SIZES (SACHS-STEFAN<sup>1</sup>).

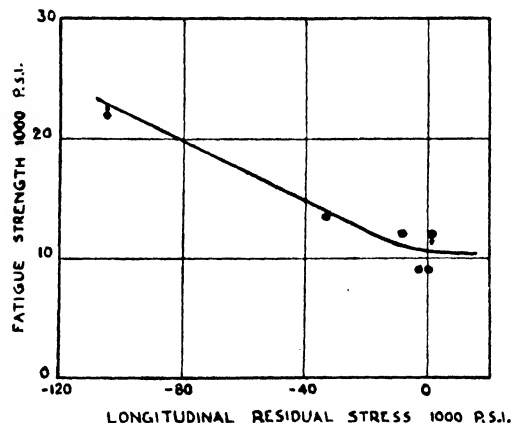


FIG. 7 — RELATION BETWEEN FRETTING FATIGUE STRENGTH AND RESIDUAL STRESSES OF 0.4 TO 0.5 PER CENT CARBON STEELS ACCORDING TO TESTS BY HORGER AND NEIFERT<sup>11</sup>.

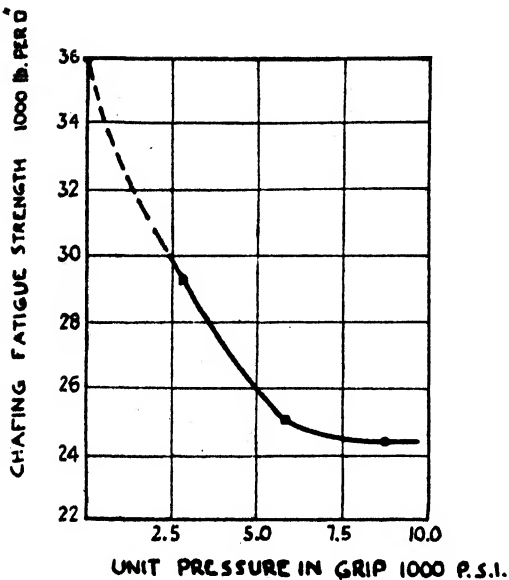


FIG. 8 — THE EFFECT OF CLAMPING PRESSURE ON THE FATIGUE STRENGTH OF CANTILEVER TYPE STEEL SPECIMENS (THUM AND WUNDERLICH<sup>33</sup>).

tude of such stresses and fretting fatigue strength is illustrated in Fig. 7.

Thin, soft inserts between and coatings of one of the contact surfaces also increase the fretting fatigue strength<sup>21,37</sup>.

The analogy between fretting fatigue and notch fatigue has been also confirmed by photo-elastic studies of contacting parts<sup>17,34</sup>. These show that the stress distribution in a loaded press-fitted part is nearly the same as that in a single piece of the same overall shape.

This explains that the fatigue strength of press-fitted parts can be raised by stress-relieving measures similar to those applied to notched (filleted) parts.

However, the fatigue strength of press fitted parts is considerably dependent upon the pressure initially present in the press fit<sup>37</sup>, as illustrated in Fig. 8.

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## Non-Technical Notes

### WATER-DETECTING COMPOSITION

WATER-DETECTING COMPOSITIONS ARE EMPLOYED to determine the depth of water layer in storage tanks or containers of petrol or vegetable or mineral oils. Hitherto such compositions were imported, but during the war, at the instance of the Defence Department, a satisfactory composition was evolved by the *Council of Scientific & Industrial Research*.

It has now been decided to make the formula available for civilian use. Considering that millions of oil containers of various types are used in the country every year, the use of this composition should be of great help in rapidly detecting the amount of water added to vegetable and mineral oils.

The composition is used by smearing it thinly on the lower portion of an immersion rod which is then dipped to touch the bottom of the oil container. The depth of the water layer in the container is indicated by the height of the smeared portion up to which colour change has occurred.

The composition is easily prepared by mixing suitable proportions of chemicals with necessary fillers, plasticizers, gum or resins, etc. Since the composition changes colour from light blue to pink red on contact with water or exposure to moisture, a close control of humidity during manufacture is necessary to obtain a satisfactory composition. The composition is packed in metallic collapsible tubes to ensure protection from deterioration by absorption of moisture from the atmosphere.

The principal equipment and laboratory apparatus required with their approximate costs are given below :

	Rs.
Edge runner mill with motor, etc., (pan dia. 26") 1 unit	5,000/-
Tube filling machine with kettle, 5 litre, 2 units	1,000/-
Laboratory equipment and apparatus	5,000/-
Dehumidifier and auxiliary equipment	4,000/-

All items of the plant and chemicals and other raw materials are easily available in India from imports or from indigenous sources.

#### Floor Space —

Mixing rooms, laboratory, etc.	
Two rooms : 16' × 16' each	512 sq. ft.
Office : one room 16' × 16'	256 "
	768 "

#### Cost of Manufacture —

	Rs.
Hydrotropic substance (1 lb.)	25/-
Filler (10 lb.)	5/-
Gum (1 lb.)	1/8/-
Plasticizer (1½ lb.)	10/8/-
	13½ lb. 42/-
Collapsible tubes, tin clad, capacity 2 fluid oz. each, @ Rs. 175/14/- per thousand, 108 in number	19/4/-
Manufacturing cost at 40 per cent of the cost of materials	24/6/-
Cost per dozen tubes containing 2 fluid oz. each of the material	9/8/3
Or net cost of 1 lb. of material inclusive of packing	6/6/-

Demand for this material is likely to be confined to specialized trade, but the cost of manufacture is low enough to justify its manufacture being taken up.



# REVIEWS

**Principles of Radar**, by Denis Taylor & C. H. Westcott (University Press, Cambridge), 1948, pp. 141. Price 12s. 6d.

THIS IS THE FIRST VOLUME IN THE SERIES of "Modern Radio Technique" prepared under the general editorship of Dr. J. A. Ratcliffe. The present volume will be most welcome to that large number of physicists, mathematicians and engineers who are interested in radar but whose equipment extends only to the fundamental principles of radio physics and technique. The volume would also be of interest to advanced students in providing them a general survey of the subject of radar as a whole.

A brief historical introduction is provided in the opening chapter. Following Appleton and Watson-Watt, a radar is defined as "a system for measuring at least two of the co-ordinates which define the position of an object with respect to the observer: one of these co-ordinates is the distance between them and is inferred directly from the time of travel of a radio wave travelling by the shortest path between them". (Readers interested more in the history of radar development will find a fascinating account in Rowe's *One Story of Radar*.) The importance of radar in peace and war has been widely recognized. Its contribution during the battle of Britain was most outstanding.

The second chapter deals with the generation and reception of pulse-modulated signals. It describes in a schematic way the magnetron, Fourier decomposition of a pulse-modulated signal, and the noise factor in a receiver. The performance of a radar equipment is described in chapter 3. The determination of range and azimuth are dealt with, respectively, in the next two chapters, and the determination of an elevation is described in chapter 6. Chapter 7 is concerned with two-dimensional scanning and air-interception radars. The separation of sea and ground clutter is discussed in chapter 8. There is also provided an interesting but all too brief discussion of the principles to be used for suppressing response from stationary targets. Electrical characteristics of some typical radar equipment are dealt with in chapter 9 and the last chapter

is concerned with I.F.F. and other secondary radar systems.

There are two appendices, one on symbols and standard formulae, and the other on the calculation of absorbing, scattering and echoing areas.

Recently there have appeared a large number of books on radar, but for any one who has little previous knowledge of the subject and who desires an elementary but serious introduction to it, the present book can be unreservedly recommended as one of the best available.

D. S. K.

**Engineering with Rubber**, by Walter E. Burton (McGraw-Hill Book Company Inc., New York), 1949, pp. xi+486. Price \$ 6.50.

"WHEN A MAN SETS UP TO BUILD A NEW product", the author goes on to say in the preface, "he may decide that some rubber parts are needed." For the sake of one not familiar with the science of rubber and potentialities of this material, the author in collaboration with the *B. F. Goodrich Company*, Akron, Ohio, has gathered material for the present book and it deals with "industrial rubber products and their applications". The book is divided into twenty-four chapters, of which the first two deal with various types of rubber, both natural and synthetic, and their fundamental properties. From rubber, the technologist fabricates myriads of products: adhesives, V-belts, transmission and conveyer belts, hard rubber goods, mountings, sponges, latex products, moulded rubber articles, etc. Rubber can be used alone or in association with fibre, fabric or metal, and the author, in the course of next twenty chapters, goes into considerable detail with regard to the product design and performance of these. One complete chapter is devoted to the use of vinyl chloride which has a place of its own in "engineering with elastomers". Strictly speaking, it should not come under the heading rubber, for it is classified under non-vulcanizable elastomers. The book ends with a chapter on case histories where the use of rubber helped in the solution of a particular design or production

problem and the author has conclusively shown that in skilled hands, rubber, like metals, is a trustworthy engineering material.

Rubber consumption statistics show that over 75 per cent of the world's production goes into the manufacture of tyres and tubes. Modern tyre is the result of painstaking research and development in the fields of textiles, chemicals and rubber, combined with a study of numerous other physical and engineering problems like rubber-metal-textile bonding, heat development on flexing, abrasion, fatigue, etc. It is surprising that no reference to this important subject is made in this book. The subject of "tyre" is big enough to be able to cover a whole volume and it is hoped that this omission will be rectified in later volumes.

There are a few inaccuracies to which attention may be drawn. It is stated (p. 9) that methyl rubber was first made around 1912 in Germany and U.S.A. While it is recognized that Germans, when cut off from all the sources of raw rubber during the first world war, developed the synthetic rubber called the methyl rubber, no such elastomer was made at that time by U.S.A. Again, it has been mentioned (p. 47) that the cements made of synthetic rubber are satisfactory for several purposes: now G.R.S. is particularly known for its "lack of tackiness" and a familiar practice is to give a coat of cement made from guayule rubber when it is desired to cement tread and carcass in the case of tyre made from synthetic rubber. The derivation of formula for centre distances in the case of pulleys worked with V-belts (p. 94) appears to be wrong. The correct derivation should be:

$$C = \frac{L - 1.57(D+d) - \frac{(D-d)^2}{4C}}{2}$$

The formula for the horse-power capacity of transmission belting should, instead of as given in the book (p. 126), be preferably expressed as:

$$H = \frac{SWP(T_1 - T_2)}{33000},$$

where  $T_1$  and  $T_2$  are the tensions on the tight side and the slack side of the belt respectively.

Throughout the book, the synthetic rubbers have been referred to as American rubbers or American made rubber. For want of a satisfactory definition, the materials developed synthetically to replace natural rubber

have been variously referred to as Butalastic elastomers, Butaprenes or more simply as synthetic rubbers, and to call these materials "American rubbers" would be tantamount to ignoring the pioneering and brilliant work of British, Russian and German scientists which lies behind the successful manufacture of synthetic rubbers of various types today.

These few drawbacks, however, do not take away the merits of the book. A strong case has been made out for the intelligent use of rubber and the present reviewer agrees with the author when he says that the customer who proposes to employ rubber should tell the manufacturer what the product is expected to do and not to specify the composition (p. 41). An almost infinite combination of properties is possible by an analysis and summing up of the service requirements and the technologist can evolve a satisfactory product. The technologist in turn can look up to the pure scientist for giving him elastomers capable of better performance and possessing properties hitherto unthought of.

M. R. VERMA

**The Application of Ionospheric Data to Radio-Communication**, by Sir Edward Appleton & W. J. G. Beynon (published by the Department of Scientific & Industrial Research, England), Special Report No. 18, 1948, pp. 44. Price 1s.

AS THE NAME IMPLIES, THE BROCHURE DESCRIBES methods by which ionospheric laboratory data may be utilized for deriving characteristics of long-distance radio wave propagation. It is assumed that the reader has the ground knowledge of the ionosphere propagation phenomena. The results are described in a manner in which they can be used by the radio engineer.

A parabolic distribution of ionization with height is assumed and it is shown how, from a knowledge of the height of maximum ionization and thickness of the parabolic layer, one can estimate the maximum usable frequency for long-distance radio communication.

The book contains mainly the works of the authors on the subject that appeared in *Proceedings Physical Society* (1944, 1947) and in some confidential papers having restricted circulation during the war. The principles detailed in this book form the basis of forecast of ionospheric propagation

conditions which are computed monthly by the Radio Research Station, Slough.

S. S. BARAL

**Microwave Transmission Circuits**, edited by G. L. Ragan (McGraw-Hill Book Co. Inc., New York), 1948, pp. xvii+725. Price \$8.50.

THE VOLUME, WHICH IS NO. 9 OF THE "Radiation Laboratory Series", deals with the theory and practice of coaxial cables and wave-guides used in the propagation of microwaves. Seven authors who worked in the M.I.T. Radiation Laboratory have contributed to it under the heads: Elementary Line Theory; Materials and Constructional Techniques; Rigid Transmission Lines; Flexible Coupling Units and Lines; Transmission Units (between coaxials and wave-guides); Motional Joints, Tuners, Power Dividers and Switches, Theory of Microwave Filters, and Design of Microwave Filters.

On account of the outward appearance of these circuits they are popularly called "plumbing". Their design is of interest not only to the microwave engineer but also to the physicist and the optical and audio engineers. This is so because at these frequencies, the cross-section of the circuits is comparable to the wavelength and diffraction and other wave phenomena become increasingly important. The components have to be machined to a much higher precision than is necessary at ordinary radio frequencies and to facilitate this the dimensions of the various components are given in inches correct to a thousandth of an inch.

Although the earlier volume, No 8, of the series, entitled *Principles of Microwave Circuits*, covers the theoretical aspects of the object, a certain amount of repetition has become inevitable in the present volume for the sake of completeness. The chapter on theory of microwave filters covers aspects applicable to low frequency waves also and it is followed by a chapter dealing comprehensively with the design of microwave filters. "The theory and techniques described in these two chapters", it has been stated, "came too late to play a significant rôle in the war, but they should prove extremely useful in peace-time applications." The term "microwave" is as vague as ever. For example, Vol. 11 on "Microwave Measurements" defines microwaves as extending from 1 mc./sec. In Vol. 8 the

word microwave does not necessarily imply a particular range of frequencies, but "a characteristic technique and aperture field". In the present volume, the term "microwave" may be assumed to include all frequencies greater than 1,000 mc./sec., but it has been specifically stated that the circuits described operate in the range from 2,500 to 25,000 mc./sec. In conformity with other volumes of the series, *mks* units are used throughout the book, except where specifically indicated; the text matter is well illustrated. The volume is beautifully got up, and is a monumental contribution to the rapidly growing literature on microwaves.

B. N. SINGH

**Research in Industry**, published by the Department of Scientific Research & Board of Trade, U.K. (H.M.S. Office, London), 1948, pp. 84. Price 1s. 6d.

THIS BOOKLET CONTAINS A SERIES OF articles written by accepted and well-known authorities in various fields of technology. These articles were originally published in the *Board of Trade Journal*, U.K., during 1947-48 and they have now been compiled into a single publication.

The first contribution is by Sir Edward Appleton on "How Science can help Industry". The other 18 articles relate to the research work carried out in various research institutions in Britain in the following subjects: cotton, glass, wool, rayon, pottery, iron and steel, electronics, lace, linen, boots and shoes, paint, furniture, the electrical industry and consumer goods, plastics, machine tools and small tools, light engineering, industrial design and radar. The articles give a brief but complete account of the outstanding researches carried out at these institutions.

The subject-matter under each head gives a brief account of the progress of research in the several branches of technology and the present trends and needs. One important feature noticeable in all these articles is the valuable contribution made by industrial research associations in the country which play so important a part in the progress of the technological research.

The pamphlet contains, in addition, 2 appendices, one detailing the establishments under the *Department of Scientific Research*, U.K., and the other is a list of industrial research institutions in Britain.

# Stablization of Plant Names\*

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ANY complaint against frequent changes in plant names is usually brushed aside by some as due to ignorance of the International Rules of Botanical Nomenclature, or a misunderstanding or a misinterpretation of those Rules, or due to a lack of experience in herbarium and taxonomic work.

The review of Kanjilal's *Flora of Assam* published in *Kew Bulletin* (1935, p. 586) illustrates the present position. The reviewer points out: "It is to be regretted that the nomenclature is not up-to-date... *Camellia theifera* Griff. (should be) *Thea sinensis* L. *Bombax malabaricum* DC. is more correctly *Gossampinus malabarica* (DC.) Alston... *Taraktozenos* has been sunk in the genus *Hydnocarpus* though there are quite adequate reasons for keeping the two distinct." It is amusing to find that in the International Rules of Botanical Nomenclature edited by Camp and others (1947), *Camellia* is given with reasons as the correct name and not *Thea*; and *C. sinensis* appears as the correct name in Sampson's *Cultivated Crop Plants of the British Empire* (published by Kew itself in 1936, just a year after publishing *Camellia* as a wrong name), and in Rehder's *Manual of Cultivated Trees and Shrubs* (1940). Most authorities now agree that the correct name for *Bombax malabaricum* is not *Gossampinus malabarica* but *Salmalia malabarica*. And *Taraktozenos* is sunk in synonymy in Burkill's *Dictionary of Economic Products of the Malay Peninsula* (1935), Hill's *Economic Botany* (1937) and many other publications. This shows what "up-to-date" means!

As contrasted with reviews which express regret at not finding the latest names (correct according to the opinion of a reviewer, but may not be so in the view of others) in publications, there is a very recent review of the 3rd edition (1948) of Dallimore and Jackson's *Handbook of Coniferae*, by W. L. T. in *Science Progress*.\* It reads: "In this edition, the old familiar specific names have been changed to comply with

International Rules: thus, *Larix europea* becomes *L. decidua* and *Pseudotsuga Douglasii* is changed to *P. taxifolia*. Some will feel regret at this surrender to orthodoxy, especially because it is undesirable that the nomenclature of species of economic value should be at the mercy of arbitrary regulations, and because under the Rules the decisions of taxonomists must always remain liable to possible further revisions."

Experiences of the following type, quoted from the first issue of *Flora Malesiana* (Ser. I, Vol. 4, Pt. 1, 1948, XVIII), are only too common when similar attempts are made to obtain authoritative opinion about the valid name for a plant. "A *Clausena* of unknown origin was cultivated for economic purposes at Buitenzorg. I referred it to *Clausena anisum-olens* (Blco) Merr. but the phytochemist was dissatisfied, the properties of the oil did not tally with data recorded from the same species in the Philippines. I then sent ample material with full notes to Dr. Tanaka, Dr. Swingle, and to the Kew and Paris Herbaria, for identification. The answers were all different and the phytochemist was, of course, disgusted with the practical results of taxonomy, because now he had the choice among 5 names for his plant."

The proceedings of the last International Botanical Congress, the papers of Furtado and others published some years back in *Garden's Bulletin, Straits Settlements*, and the recent symposium on plant nomenclature (which was arranged in view of the forthcoming International Botanical Congress, and published in *Amer. Journ. Bot.*, 1949) illustrate the defects in our Rules and nomenclature. The International Rules of Botanical Nomenclature are undoubtedly made to bring about stability of plant names. But if confusion prevails, apparently on an increasing scale, in spite of the Rules, and names published as correct by individual botanists are later shown to be wrong, it is time to review our "Rules" as well as our approach to the problem. The proof of the efficacy of the Rules is

\* 1949, 37, 378.

\* See also *J. Sci. Ind. Res.*, 1949, 8, 163.

the uniformity in the publications of experts who have worked at big herbarium centres, and followed the Rules of Nomenclature in determining the correct name of a plant.

Burkill, for his *Dictionary of Economic Products of the Malay Peninsula* (1935), and Sampson, for his *Cultivated Crop Plants of the British Empire* (1936), utilized to the fullest extent the resources of the Royal Botanic Gardens, Kew, and both are avowed followers of the International Rules. But it is surprising to find that for a very large number of plants, different names are given as valid by them. A comparison of Hill's standard work on *Economic Botany* (1937) and Holland's *Overseas Plant Products* (1937) shows a similar deplorable difference. In many cases, the valid name of one is the invalid name of the other! Both of them cannot be correct. Both these authors are also followers of the International Rules and, like Burkill and Sampson, very careful workers. Then why this difference? Who is correct and for which name? Or compare again the names given by Reeder in his monograph on *Grasses of New Guinea* (*Journ. Arnold Arboretum*, 1948) with those given by Bor, an acknowledged authority on Indian grasses, in Vol. V of the *Flora of Assam* (1940). Reeder has revived a number of old synonyms to validity, and reduced the valid names in Bor's work to synonymy. Or, compare some of the names given in *Official Guide to the Museums of Economic Botany*, No. 1, Kew (1930), with those in *Hand-list of Tender Dicotyledons*, Royal Botanic Gardens, Kew (1931); or the names in Sampson's book with those given in Hill's. These are just a few examples to indicate that, at present, stability

or uniformity in plant nomenclature is a myth. It shows that something is fundamentally wrong with the approach to the nomenclatural problem. It is the realization of this utter confusion, attended with too many and too frequent changes, that prompted the International Botanical Congress to appoint a committee to draw up a list of economic plant names which is to remain in force for a period of 10 years.

I wish to make it clear that examples can obviously be given from the writings of systematic and economic botanists. It is in the nature of things and the present state of the subject that discrepancies in nomenclature occur, in spite of the best efforts of writers to find out correct names. The examples given do not reflect anything on the writers but merely indicate that there is an urgent need to stabilize plant names by some means or other. Let wrong names be changed, by all means; but let the change be to the correct one, and not to another wrong name. Rehder's article on Names and Nomenclature in Bailey's *Standard Cyclopaedia of Horticulture* contains the following emphatic statement:

"Whenever all botanists of authority agree upon a set of changes, the horticulturists should of course adopt them; but a cyclopaedia of this kind is not under obligation to be 'up-to-date' in respect to recent changes that may represent only individual opinion, or which are likely to be overturned or at least not adopted by other botanists of standing. So far as possible a cyclopaedia of horticulture should represent settled practices. This work, therefore, adopts a conservative attitude in nomenclature."



# Silk Production & Weaving in India<sup>1</sup>

**T**HE monograph, *Silk Production & Weaving in India*, just published by the Council of Scientific & Industrial Research, is a comprehensive treatise dealing in detail all aspects of Indian silk industry as it exists today. The subject-matter is distributed over 11 parts, each part giving a brief but a complete account of the subject under its head. The monograph contains 2 important appendices, one dealing with how Japan developed her silk industry and the second refers to lines of research in sericulture, both of which are of special interest to research workers and may be read with much profit.

Part I of the monograph is in the nature of a general survey of silk industry and its requirements. Of interest to the reader and to one interested in silk industry are the short accounts under the following heads: the present state of silk industry in India, essentials of a successful silk industry and the future of silk products. The next 5 parts deal with mulberry, silk-worm, rearing of silk-worm, reeling and raw silk. Part VII deals with by-products of silk industry. All aspects of *eri*, *muga* and *tasar* silks and the lines on which research is necessary under each head are given in the next 3 parts. The last part under the title "Silk Weaving and Other Manufactures" deals with variety of subject such as raw materials in weaving, looms, types of manufacture and manufacturing processes, standardization of silk fabrics in Japan; production of standardized silk fabrics in India, etc. No aspect of silk and silk industry is omitted from this exhaustive monograph. Its value is apparent from the fact that the author has collected valuable factual data province by province and state by state to present a very clear picture of the future possibilities of the industry. The indigenous methods of silk-worm cultivation and silk production have been critically compared to the conditions obtaining in countries like Japan where silk industry has almost reached perfection. Fully illustrated with 90 useful and informative drawings, and reference tables containing exhaustive statistics on every aspect of silk industry, the publication is sure to be a valuable addition to the libraries of all technological institutions.

Of the research aspects dealt with in the monograph, the following invite attention:

**Eri Silk** — Castor is the staple feeding material for the *eri* silk-worm. There are many indigenous varieties of castor and selection of suitable varieties with respect to their growth, yield and food value of the leaves, effect of soil and climatic conditions and yield of seed has to be made. *Heteropanax fragrans* (*kesere*) has to be tried for its suitability as a food plant. Diseases of the worm in relation to climate and food and a study of the worm with a view to have the best cocoons are the other problems to be investigated.

**Muga Silk** — Investigations are to be initiated on the following: to find out if there is more than one species of *muga* worm; relative values of known food plants of the worm; experimental cultivation of *sum* and *hualu* on a plantation basis, occurrence and investigation into causes of *flacherie* and *grasserie* diseases in the worm; the worm

produces creamy-white coloured silk when grown on *meazankuri* and *champa* unlike the common golden-coloured silk when fed on *sum* and *hualu*. The former fetches high prices and investigation into the fact that age of trees as well as the quality of leaves influence the type of silk produced can be taken up with profit; there is considerable scope for improvement in the reeling process of *muga* cocoons; a process for giving twist to the yarn as it is reeled has to be developed; methods for easy rearing and increasing the yield of silk have to be developed.

**Tasar Silk** — A systematic study of the various types of *tasar* silk-worms has to be undertaken to enable easy identification of the varieties. Methods to bring about mating of the worms under domesticated conditions have to be developed. Difficulties encountered in rearing of the worms could be minimized if steps are taken to characterize, isolate and supply seed cocoons of definite voltine characters. Cultivation of *muga* worm on a plantation basis using *asan*, *arjun* and *sal* plants has to be tried on a field scale. Reeling of *tasar* cocoons needs investigation in order to evolve suitable machinery and methods on the same lines as in the case of *muga* silk.

**Research in Sericulture** — There has been hardly any research on sericulture in India though its necessity has been felt and stray attempts have been made. It has not been possible to date to acclimatize foreign mulberry varieties to conditions obtaining in silk-producing areas in the country. Cocoons produced in Bengal are of a poor grade and intensive research is necessary to improve their quality. Kashmir imports univoltine French eggs; attempts to produce them locally have failed. Development of hybrid races have helped Mysore silk sericulturists to obtain better cocoons. Work on this line has to be extended to other centres.

The main lines of research on mulberry, silk-worms, diseases of silk-worms are:

**Mulberry** — Selection of varieties after a systematic study of indigenous and foreign varieties; study of the various varieties with special reference to chromosome number, biochemical aspects, draught resistance and genetical studies with reference to inheritance characters; study of soils in relation to the growth and health of mulberry; and physiology of mulberry plants.

**Silk-worms** — Acclimatization of suitable univoltine worms in India; hybridization to secure improved cocoons; genetical studies, physiology and relation of the life of the worm to climatic conditions; rearing of cocoons having high silk content and high reability index and minimum boil-off loss; the uniformity of the cocoons is a very important factor in obtaining the above results; analysis of silk fibre from different races; physical studies of the silk fibre and development of economical methods of rearing.

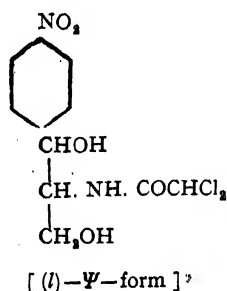
**Diseases of Silk-worms** — The physiological diseases of silk worms, viz. *flacherie*, *grasserie*, *gattine* and *court* are not properly understood and require investigation. A useful lead can be had from the fact that these diseases are primarily due to disturbances in the metabolic or normal physiological functions of the worm by climatic factors and through deficiency or excess of certain elements in its food.

\* *Silk Production & Weaving in India*, by C. C. Ghosh, C.S.I.R., Monograph, 1949. Price Rs. 8.

# NOTES & NEWS

## Chloromycetin Synthesis

THE STRUCTURE AND SYNTHESIS of chloromycetin have been recently described in a series of papers originating from the laboratories of Parke, Davis & Co. (*Nature*, 1949, 163, 553). The empirical formula,  $C_{11}H_{12}Cl_2N_2O_6$ , has been assigned to it and it is shown to form a di-*O*-acetyl compound. Acid or alkaline hydrolysis produces a halogen-free base and a halogen-containing acid. The base contains both nitrogen atoms, one of which is present as a primary amine. Chloromycetin is described as (*l*)- $\Psi$ -1-*para* nitrophenyl-2-dichloro acetamido propane-1:3-diol and has the following structure:



The molecule contains 2 asymmetrical carbon atoms and only 1 of the isomers is identical with the natural compound and the other 3 are biologically inactive. The synthetic chloromycetin appears to possess the same rickettsiostatic and virustatic properties in experimental infections and the same usefulness in treating patients with scrub typhus.

## Synthesis of *p*-Aminosalicylic Acid

A NEW SYNTHESIS FOR *p*-AMINOSALICYLIC acid, a drug which has potential application in the treatment of tuberculosis, has been developed at the Research Laboratories of Hoffmann-La Roche, Inc., New Jersey (*Phar. Assoc., Scientific Ed.*, 1949, 9, 38). 4-nitro-2-chlorobenzoic acid is the key intermediate. 4-nitro-2-aminotoluene is converted to 4-nitro-2-chlorotoluene by the Sandmeyer reaction. This latter compound is oxidized

to 4-nitro-2-chlorobenzoic acid by an improved procedure using 80 per cent sulphuric acid and potassium dichromate. The action of carbonate-free calcium hydroxide on this chloracid yielded 4-nitrosalicylic acid and gave *p*-aminosalicylic acid with an over-all yield of about 40 per cent when reduced with stannous chloride in concentrated hydrochloric acid.

## High-pressure Acetylene Chemistry

THE FIRST PILOT PLANT IN THE U.S.A. for the manufacture of acetylene derivatives, employing high temperatures and pressures, which not many years ago were considered to be impracticable and hazardous, has been put into operation by the General Aniline Co. (*Chem. Age*, 1949, 60, 625). It represents the first development employment in the U.S.A., with considerable amplification, of the principles originally developed by the I. G. Farbenindustrie chemist, Dr. J. Walter Rappe. With this development, a new field of organic synthesis has opened up which should prove full of possibilities in the manufacture of adhesives, pharmaceuticals, paper, rubber, textiles, etc.

By making use of Rappe's discovery that acetylene could be handled under pressure and at elevated temperature if the gas streams were divided into smaller flowing streams, it was possible to increase the use of acetylene as a chemical raw material. New products from this source that now appear to be readily available and of considerable potential industrial use are vinyl ethers, butynediol, propargyl alcohol, and many others. Preliminary studies showed that from these intermediates interesting resins, adhesives and rubber-like polymers could be made.

The technique permitting the safe use of acetylene under high pressures consists of 2 methods, one involving the dilution of acetylene with an inert gas and the other wherein acetylene was reacted in small-bore equipment providing a minimum of free space for gases to collect. In its new plant in the General Aniline Co., extensive instrumentation

with remote controls has reduced the explosion hazard to a minimum.

Vinylation and ethylenation are 2 processes employed in the synthesis work. The vinylation products include methyl, ethyl, butyl, and iso-butyl vinyl ethers. The new technique of diluting acetylene with an inert gas has been employed in order to carry out reactions with low-boiling alcohols.

Condensation of formaldehyde and acetylene was carried out using acetylene under high pressure and temperature and employing a most unusual catalyst, copper acetylide; propargyl alcohol and butanediol were some of the products of this ethylenation. Special equipment and techniques are employed in this process as the dilution method was not suitable.

## Chrome-yellow Manufacture

CONTINUOUS PROCESSES FOR THE manufacture of chrome yellow and molybdate orange and a semi-continuous process for manufacture of zinc yellow have been recently developed at E.I. du Pont (*C.T.J.*, 1949, 124, 425). The production time for chrome yellow is only about 2 hr. from receipt of raw material to bagging of finished product.

Lead nitrate and sodium chromate solutions (35 per cent) are allowed to react in a specially designed precipitation reaction chamber, which is very small compared to continuous precipitation tanks and in which the precipitated mass remains for a few seconds only. The amount of solutions let in are controlled automatically. The lead chromate slurry is fed off to a series of vessels in which the temperatures are controlled to give the desired particle size temperatures and pH of the slurry is automatically controlled. The slurry is centrifuged in stainless steel Bird centrifuges (washed during centrifuging) and dried in a Proctor and Schwartz dryer assembly. The yield is 99 per cent of the theory.

A similar process is used for the manufacture of molybdate orange, but in the zinc-yellow process the actual precipitation and particle size development stages are carried out batchwise. The zinc yellow is a basic potassium zinc chromate, and the reaction is carried out in large agitated tanks. The same finishing plant is used



for all the pigments and has, therefore, been designed for easy cleaning.

The new process turns out yellow pigments having higher tinting strength, better light fastness and working properties as well as better stability and uniformity of particle size than was obtainable with the old methods of production.

### A New Method for Determining the Weight of Cellular Structures

SUBSTITUTION OF A CONTINUOUS spectrum for monochromatic X-rays in the methods now being used for cytochemical elementary analysis makes it possible to weight single cell structures when a sufficiently broad band of a continuous X-ray spectrum is used. The effects of the absorption jumps of the elements in the tissue can be neglected, and the total absorption is proportional to the total mass of tissue. The wavelengths of the chosen band must be selected properly.

A thin collodion foil ( $c. 0.3U$ ), laid over the slit in the metal preparation holder serves as supporting membrane for the preparation. The microtome section or a smear of the tissue being examined is laid on one half of that membrane. The other half of the membrane is reserved for an absorption wedge made of thin collodion foils. The X-ray absorption of the foils should be of the same order as that of the sample. The preparation is laid against the photographic emulsion of a Lippmann film or any other very fine-grained photographic emulsion. Very good contact must be secured. A micro-radiogram is registered with long wavelength continuous X-rays (500-4,000). The voltage chosen depends on the nature of the sample. The X-ray image of the sample and the wedge is enlarged 200-500 times by photomicrography. Absorption in single cell structures can be compared with the absorption in the collodion foils, and the relative distribution of the amount of dry substance in the sample can be computed by photometric measurements on the enlarged image.

Knowing the weight per unit surface of the collodion foil and introducing the correction factor, the absolute weight per unit surface of the cell structures can be calculated. It is possible to weigh structures as small as  $1\mu$  in diameter, which is the resolving power of the Lippmann film.

This method is now being used in combination with X-ray methods for quantitative estimation of single cell elements (*Nature*, 1949, 163, 563).

### Thickness Measurement by Beta Emission

THE PRINCIPLE OF THE CONTINUOUS measurement of the thickness of sheet materials is demonstrated by a new instrument called the beta-ray thickness gauge recently announced by the *General Electric Co.* (*Chem. Age*, 1949, 60, 557). By measuring absorption, the device indicates the mass per unit area of the material under test, but can be calibrated in terms of thickness to help operators maintain product uniformity, reduce the amount of rejected material, and save on the amount of raw material used. The new gauge is expected to find application in keeping check of the thickness of metal foils such as aluminium, copper, tin brass and steel, being rolled at high speeds. It can also be employed with plastics, textiles, rubber and other sheet materials, especially those that cannot be contacted because of their condition while in processing.

In operation, the beta-ray thickness gauge measures the deviation from a chosen setting by registering the amount of  $\beta$ -rays which the material under test absorbs. The source of  $\beta$ -rays in the gauge is 2.5 millicuries of strontium 90. Those rays, unabsorbed by the material passing through the gauging head, are gathered in an ionization chamber. An attenuated 90-cycle signal is added in phase opposition to cancel the signal from the ionization chamber. The attenuator voltage, therefore, is a measure of the ionization chamber voltage, and of the amount of material in the  $\beta$ -ray beam.

Operating on a power supply of 100-125 volts, 60 cycles,  $\pm 0.3$  cycles, the power consumption of the gauge is about 150 watts. Its accuracy is said to be  $\pm 2$  per cent or better between normal periods of calibration, while drift is not more than 1 per cent per hour after a 30 min. warming-up period. Under normal conditions it is claimed that calibration need not be made more often than once every 4 hr.

### Evaluation of DDT

BIO-ASSAY PROCEDURE OF EVALUATING the potency of DDT and its preparations are intricate and the

conditions of test experiments are difficult to define. Chemical methods of measuring DDT potency described recently (*Scientific Agriculture*, 1949, 29, 53) enable accurate definition of one of the variables influencing the bio-assay methods of evaluation — the amount of insecticide to which the insects are exposed.

Three methods are described: (1) gravimetric methods; (2) volumetric methods; and (3) colorimetric methods. The gravimetric methods are especially useful for assay of technical grade DDT and DDT dusts, where  $p,p'$ -DDT crystallizes out after refluxing the sample with a 75 per cent solution of ethanol saturated with  $p,p'$ -DDT. The volumetric methods depend on the estimation of the labile chlorine in DDT. Of the 5 chlorine atoms in DDT molecule, one is detachable by hydrolysis in the presence of alkali. The labile chlorine atom, having become inorganic chlorine, is then measured by the Volhard titration method or electrometrically. Estimations based on total chlorine content are not specific for DDT.

The colorimetric method, using xanthidol-pyridine-potassium hydroxide reaction, is rapid and sensitive and is adoptable for qualitative and rough quantitative field work. In a second method, the Schechter-Haller method, DDT is intensively nitrated, the isolated tetra-nitro DDT is dissolved in benzene and to which methanolic sodium methylate is added when a blue colour results showing a maximum absorption at  $600\mu$ , the intensity of which is proportional to the amount of  $p,p'$ -DDT. This method can also be employed for the estimation of  $o,p'$ -DDT which gives a violet-red colour with 2 absorption peaks at 590 and  $510\mu$  respectively. This method is specific and sensitive for  $p,p'$ -DDT.

### Refining of Oils Containing Vitamin A

MOLECULAR DISTILLATION OF VITAMIN A rich oils, while yielding concentrates of a high vitamin A content, requires, in order to obtain satisfactory results, oils free from impurities and with low acid values. Alkali refining is shown to yield good, refined oil and the neutralized oil, far from losing anything of its original potency, has an increased potency (*Nature*, 1949, 163, 722). In oils with high potency and high acid value, this increase can be quite substantial.



A sample of Groper liver oil with 120,000 i.u. of vitamin A per gm., 12.5 per cent free fatty acid and 3.95 per cent impurities, after neutralization with an excess of sodium carbonate, centrifuging and filtering, gives a refined oil (76.5 per cent yield) with a potency of 140,000 i.u. of vitamin A per gm. The loss due to oil stock trapped in the soap can be minimized employing the latest refining methods. Thus the alkali-refining method provides an economical and simple method of purifying oils containing vitamin A.

### Treatment of Leather with Synthetic Resins

METHODS DEVELOPED FOR TREATING leather with synthetic resins are described (*J. Res.*, 1949, 42, 63). Acrylate monomers have excellent penetration and can be polymerized *in situ* to give increased wear resistance of sole leather and water resistance to upper leather. The acrylates and methacrylates are studied because of their ready availability and relative ease of polymerization.

Three methods are employed: (1) immersion of leather in monomer and subsequent polymerization *in situ*; (2) immersion in polymer solution and followed by evaporation of solvent; and (3) immersion in partially polymerized resin followed by varying degrees of cure depending upon the desired results.

In the first method, leather specimens are immersed in a monomer containing benzoyl or lauroyl peroxide catalyst for 15 min., wiping of excess solution and heating the samples in a closed system overnight at a given temperature. The use of polymer solutions in which the molecular weight of the resin and the viscosities of the solution is low is a necessary condition in the second method. The solutions are prepared by refluxing a solution of the monomer with a lauroyl peroxide catalyst until the desired solids content is obtained. The leather specimens are immersed for half an hour, the excess wiped off and the solvent evaporated for 4 to 6 hr. The third type of resin solution that holds out promise for treating sole and shoe upper leathers is a polysulphide rubber dissolved in toluene. A proprietary preparation, Thiokol LP-2, has been employed; using acrylate polymers as much as 50 per cent resin can be incorporated in vegetable-tanned crust leather.

In the case of specially prepared solution polymers, impregnation up to 25 per cent resin has been obtained in vegetable-tanned crust, and 50 per cent in chrome retanned upper leather. Treatment with Thiokol LP-2 can be controlled to give a stiff sole or to produce a soft shoe upper leather.

Abrasion resistance of vegetable-tanned crust leather is improved about 75 per cent by *n*-butyl methacrylate polymerized *in situ*. Treatment with Thiokol LP-2 improves abrasion resistance by about 30 per cent, whereas solution-copolymers of *n*-butyl methacrylate and ethyl acrylate show no improvement. Half-hour water absorption data on vegetable-tanned crust leather indicates that treatment with *n*-butyl methacrylate, polymerized *in situ*, show 95 per cent reduction over untreated leather. Water vapour permeability of degreased chrome-retan upper leather is reduced by 60 per cent, but the reduced value of about 400 g/m<sup>2</sup>/24 hr. is considered ample for foot comfort.

### New Plasticizers

SEVERAL NEW PLASTICIZERS HAVE been described (*Chem. Age*, 1949, 60, 548). Of these di-*iso*-octyl adipate, dibutyl "cellosolve" adipate and dioctyl adipate are the latest additions.

Di-*iso*-octyl adipate is a primary plasticizer for most resins and imparts permanent flexibility, low water extractability, extremely low temperature flexibility, good electrical properties and heat stability, excellent stability to ultra-violet light, and easy processing. It is a very efficient plasticizer and produces plastic films with low plasticizer-to-resin content with good clarity and good handle, drape and resilience. Di-*iso*-octyl adipate is particularly suitable for use with nitrocellulose and produces clear, elastic films. With ethyl cellulose it gives good quality, clear, elastic films.

Films of polystyrene plasticized with di-*iso*-octyl adipate are pliable, non-tacky and practically free from blocking. It also produces good temperature-flexible stocks with synthetic rubbers. Di-*iso*-octyl adipate has a freezing point of -65°C., a flash point of 186°C. and a fire point of 236°C. At 25°C., this plasticizer has no solubility in water, but it is completely soluble in petroleum and mineral oil.

Dibutyl "cellosolve" adipate (dibutoxyethyl adipate) has a

freezing point of -30°C., a flash point of 188°C., and a fire point of 277°C. It has found wide use in the production of safety glass because it imparts excellent ultra-violet light stability and extremely low temperature flexibility to the plastic sandwich layer between the two glass plates. There are also other valuable properties of this compound which come into play when used in the combining of paper or textiles. Dioctyl adipate has a freezing point of -70°C. (clear gel) and a pour point -69°C., a flash point of 192°C. and a fire point of 299°C. and mid-boiling-point at 4 min. of 213°C. It is a very efficient plasticizer and produces good elastomers with low plasticizer-to-resin content. By comparison it has higher solvent power and it is more efficient than dioctyl phthalate in a ratio of 29 per cent to 33 per cent.

New in this field of fatty acid esters are tetrahydro-furfuryl oleate, which possesses superior internal lubricating properties, and diethylene glycol dipelargonate for low temperature performance.

The dibasic acid, azelaic acid, is now being used as a raw material for the production of plasticizers. It is also recommended for the manufacture of non-drying type alkyds, where it replaces a portion of phthalic anhydride and results in greater flexibility, improved toughness, good adhesion and good colour. The esters of azelaic acid are likely to be used to an increasing extent where high efficiency, excellent low temperature performance, negligible volatility and good oil resistance are needed for vinyl plastics.

The 2-ethyl hexyl ester of azelaic acid imparts low temperature flexibility to films which are clear, free from haze and possess excellent tear strength. Another interesting azelaic plasticizer is di-octyl azelate which is of value for plasticizing polyvinyl chloride and polyvinyl acetate-chloride cast films and calendered sheet.

Alkyl carbonates of lactates produced by acetylating lactic esters with alkyl chloroformates are newcomers to the range of lactic acid plasticizers. All these esters are compatible with ethyl cellulose and polyvinyl chloride, and judged by modulus and brittle point, these new lactates are more efficient than 2-ethylhexyl phthalate.

The esters of aconitic acid are particularly valuable for the vinyl polymers where chemical stability of highest order is required.

Polymeric plasticizers based on aconitic acid possess extremely low volatility and very low extractability by water and oil.

Ranking high in phosphate plasticizers is "Santicizer 141" produced by *Monsanto*. This is an alkyl aryl phosphate which possesses low toxicity, high compatibility with vinyl resins, low volatility, softness and drape, resistance to weathering, strength, elasticity, abrasion resistance, good low temperature flexibility. By incorporating this phosphate plasticizer in polyvinyl chloride compounds, it is possible to achieve exceptional flame-retarding qualities without sacrificing flexibility or drape. Cresyl diphenyl phosphate is another in this group with improved low temperature flexibility and superior light stability.

The glycol esters are good, all-round plasticizers, and of these triglycol dihexoate, triglycol dioctate and polyethylene glycol di-2-ethylhexoate are of immediate interest. The last-named promises to find many applications because of its unique properties. It shows the same order of non-volatility from compositions of vinyl chloride-acetate compositions as does dioctyl phthalate and, in addition, it is very efficient, since only 30.5 per cent is required to produce standard elongation.

To those working with surface coatings, moulded, extruded, and calendered plastic compositions and synthetic rubber, the polyethylene glycol di-2-ethylhexoate is of considerable value.

Nitrite rubber, a copolymer of butadiene and acrylonitrile, is another non-migratory plasticizer of special value for vinyl films to be used for food packaging. Its high molecular weight enables it to resist extractions by edible fats and greases.

### Pliofilm

"PLIOFILM", A TRANSPARENT rubber hydrochloride film, is being produced on a commercial scale by the *Good Year Tyre & Rubber Co.* (*Chem. Age*, 1949, 60, 618). The material has many potentialities as a protective packaging material.

Crude natural rubber of good grade is milled and dissolved in benzene to obtain a cement containing 20 per cent solids. The material is cooled with refrigerated water and predetermined amounts of dry hydrochloric acid gas is let in and the rubber is allowed to continue ageing for 4-6 hr.

After compounding, the pliofilm cement is filtered twice and transferred to aluminium storage tanks maintained at an elevated temperature to keep the cement from solidifying. After a third filtration, it is made into a film of 0.01" thickness.

One of the merits claimed for pliofilm is its capacity to retain or repel moisture according to needs; it is considered to be moisture-vapour-water-proof. It has also been found to be resistant to punctures, tears and abrasions, pliable though it is. It is unaffected by atmospheric conditions or normal temperatures and is not harmed by the rigorous processes of forming packaging material into a finished package. It is also resistant to weak acids and alkalis, to grease, oils, moulds and vermin. It is non-explosive, non-inflammable, odourless and tasteless.

Pieces of pliofilm can be joined by heat sealing, the resulting weld being twice as strong as the original material.

One of the unusual characteristics claimed for it is its ability, when drawn under tension to a thin membrane, to hold in moisture and retain gases such as carbon dioxide, to slow down the metabolism of wrapped fruit. Pliofilm has been successfully used to contain and preserve frozen foods, meats, cheese, coffee, tobacco, and liquid and semi-liquid products including oils.

### Bright Aluminium Alloys

AN IMPROVED METHOD OF BRIGHTENING aluminium alloy surfaces is claimed in an English patent with special reference to making the brightening more permanent and less affected by atmospheric tarnishing or other adverse action (*Chem. Age*, 1949, 60, 690).

The surface is first thoroughly degreased (by electrolytic methods), then immersed in strong acid (*pH* below 1) solution of phosphoric and chromic acids and water for 30-60 min. at a temperature of 80°-90°C. This produces a uniform surface, whether it has been previously polished or not, and serves to descale, removing any oxide without attacking the metal itself. The surface is then subjected to anodic oxidation in an alkaline solution containing trisodium phosphate and sodium phosphate or aluminium phosphate and caustic soda. The *pH* should not be lower than 11.

The part to be brightened is made the anode; temperature is

70°-95°C., voltage 8-20 v.; current density 4-10 amp./dm.<sup>2</sup>; and time required is 5-10 min. By this means a thin coating of aluminium hydroxide is deposited and is followed by rinsing with running water. The film formed, at first in a powdery condition, is dissolved in a solution of phosphoric and chromic acids, similar to that used before. Colouring with organic dyes may be imparted, and surface finally sealed with boiling water or by other means.

### New Uses for Cotton

MODERN RESEARCH HAS EXTENDED the uses for cotton in many fields. In plastics, cotton is being employed as the base for plastic products. Cotton seed hulls, hitherto considered a waste product, is finding increasing application in plastic industry.

Inexpensive cotton fabric that could be made without the usual spinning and weaving processes are visualized in the near future. Cotton fibres bound together with a thin layer of plastic application forms an unspun, non-woven fabric suitable for many types of products. Among the unspun articles now being marketed are towels, napkins, draperies, etc. These articles, though inexpensive enough to be discarded in a short while, last longer than the usual cotton fabrics and have a softer and more luxurious appearance.

Coated cotton material resembling rubber and possessing many advantages over leather is being developed and used for upholstery, luggage, and shoes. The material is stain-proof, dust-proof and waterproof. Mixed with asbestos fibre (20 per cent) the resultant fabric has excellent water absorption and polishing properties.

Combined with aluminium and glass yarns, unusual fabrics have been developed. A new protective cotton fabric is made to reflect light which can be seen by night motorists at a distance of half a mile. A variety of cotton gauge developed is capable of being absorbed in the human body within a few weeks (*USIS*).

### New Cleansing Agents & Disinfectants

DEVELOPMENT OF A POWERFUL series of "multi-cleanser" soap substitutes, which kill germs instantaneously besides removing dirt, has been reported to the *American Chemical Society* recently. The new detergents can be used for

surgical instruments and household utensils alike. These products, known chemically as "morpholinium alkyl sulphates", are so potent that a teaspoonful of detergent is enough to yield a gallon of suds, and at the same time to match the bactericidal strength of a pound of carbolic acid. These do not require caustic substances or metallic salts to do their work and have several advantages over soap; they do not break down and form insoluble deposits or cause the familiar "bath-tub ring" when used with hard water. Since they are non-metallic, they do not leave the caustic, slippery feeling on the skin which follows the application of soap. These non-metallic and non-caustic products described in the report show remarkable chemical stability and compatibility with other substances encountered in household use and in certain industrial processes (USIS).

### Softening of Hard Water by Aeration

SEMI-LARGE-SCALE EXPERIMENTS on the softening of hard waters by aeration in the presence of added precipitation chalk conducted by the *Water Pollution Research Board, D.S.I.R.*, London, have shown that it is possible to reduce the alkalinity of deep-well water from about 255 parts of calcium carbonate to about 70 parts per million when diffused air at a rate of about 10 litres of air per litre of water per hour for a period of 5 or 6 hr. is passed. The presence of finely divided chalk in suspension in a concentration of about 1,000 parts of calcium carbonate per million is required if softening is to proceed at a reasonable rate; use of greater amounts gave no further advantage. To maintain the chalk in suspension agitation other than that provided by the diffused air has been found to be necessary. Repeated use of the same chalk in 10 successive experiments did not cause any reduction in the rate of softening obtained, although a slight increase in the size of the particles of chalk was observed.

### Biological Removal of Sulphate from Water

A BIOLOGICAL METHOD FOR THE removal of sulphates present in bore-well water and render it suitable for irrigation purposes has been worked out by the *Water Pollution Research Board, D.S.I.R.*, London. Bore-well water con-

tains total solids of the order of 3,000 p.p.m. of which the major portion is generally calcium sulphate. Sulphate-reducing bacteria are employed for the purpose, the reduction occurring under anaerobic conditions. Oxygen is completely excluded and provision for the continual removal of hydrogen sulphide is also necessary.

A rectangular reaction vessel, with a capacity of about 1 gal. and having internal baffles giving in effect a passage of about 40" long and 2" by 3" in cross-section, packed with degreased steel turnings and maintained at 30°C., and filled with iron turnings was used. Oxygen-free air was supplied.

Of the various media employed, the one selected contained sodium lactate 5 gm., dipotassium hydrogen phosphate 0.5 gm., ammonium sulphate 2 gm., and water 1 litre. The medium was not sterilized but was freed of oxygen by flowing in a rapid current of carbon dioxide. The reaction vessel was filled with settled sewage and stored for several days to provide an inoculum of suitable bacteria. The sewage was then displaced by the medium at a rate of about 120 c.c. per hour, giving a period of retention of about 36 hr. The water to be treated was let in next with the nutrient salts. On the 13th day about 25 per cent of the sulphate present was removed and by the 25th day the effluent was free of sulphate. The pH fell rapidly after the 63rd day and a new batch of medium had to be introduced with the pH value adjusted to 6.5.

These experiments indicated that sulphate in concentrations approximately equivalent to that in a saturated solution of calcium sulphate could be completely removed from water at a sufficiently high rate.

Experiments designed to replace lactate by grass extracts (by steeping grass in water for a week) showed a considerable reduction in the concentration of sulphate after the 87th day of continuous running, the sulphate content being reduced from 4,000 p.p.m. to 1,000 p.p.m.

### Sand-lime & Concrete Bricks

THE *Building Research Station, D.S.I.R.*, London, has published a bulletin (*National Building Studies, Bulletin No. 4, H.M.S.O. Office, London; price 6d.*) which gives a brief account of the properties of sand-lime and concrete

bricks, and amplifies the information given in earlier reports.

The principal materials used in making sand-lime bricks are siliceous sand, lime and water, the proportion of lime ranging from 5 to 9 per cent. Hydration of lime must be complete before the bricks are pressed and limes must be high calcium limes and reasonably pure.

Ground quicklime and sand with the requisite quantity of water are thoroughly mixed in a screw or pan mixer and compressed under high pressure in a rotary table type press and steam cured for 4-15 hr. using high pressure steam (120-195 lb./sq. in.).

For concrete bricks, Portland cement is usually employed. Most of the concrete bricks are made with a dense natural aggregate; furnace clinker, blast-furnace slag and clay brick waste are also used. Clay brick waste or rubble should not contain more than 1 per cent of acid-soluble phosphate. The proportion of cement to aggregate in concrete bricks with dense aggregates varies from 1:5 to 1:12 by volume according to the type of aggregate, moulding process and quality of brick required. Concrete bricks harden and develop their strength without any artificial curing.

The strength of dry sand-lime and concrete bricks is from 30 to 50 per cent higher than when saturated. The compressive strength for sand-lime bricks is of the order of 100-5,000 lb./sq. in. and for concrete bricks with natural aggregates, 1,000-6,000 lb./sq. in.

Under normal conditions of exposure, good sand-lime brick lasts well; inferior quality has poor weather resistance and should not be used for external work. With ageing they improve in strength. Exposure to salt spray leads to erosion of the brick. Good quality, 9" sand-lime brick satisfies the 6 hr. fire resistance test in all respects.

It is preferable that both sand-lime and concrete bricks should be used in a reasonably dry condition in order to minimize drying shrinkage effects.

### Prevention of Dazzle on Roads

THE *Road Research Laboratory, D.S.I.R.*, London, has investigated into the problem of head-lamp dazzle on roads and the results have been incorporated in a recent technical paper (*Road Research Technical Paper No. 14, H.M.S.O. Office, London, price 6d.*).

Of the two measures to deal with the problem, viz. (1) those which might be applied to existing lamps with promise of immediate improvement; and (2) use of polarized light or improved lighting systems; the former has been dealt with in the paper. The paper deals with: nature of the "single beam" and "multiple beam" systems; present state of the law relating to head-lamps; a study of drivers' behaviour and the prevalence of dazzle; action recommended in relation to existing vehicles; proposed rules for the adjustment of lamps; and education of the motorist. The two appendices deal with: instructions for adjusting head-lamps and pass-lamps, and a section entitled "Some additional conclusions to be drawn from the dazzle survey".

The survey reveals that most of the dazzle on roads is due to dipped head-lamps and pass-lamps, the low-mounted pass-lamps being especially liable to this failing. It is recommended that lamps should be set so that driving beams do not rise above the horizontal and passing beams are dipped at least 3 degrees. It is also recommended that head-lamps and pass-lamps should be mounted not higher than 3' 6" and preferably not lower than 2' 6" but never less than 2' from the ground.

### Hand-operated Threshing Machine

A HAND-OPERATED MACHINE which can thresh nearly 1 cwt. of wheat, oats, barley or rice in 1 hr. has just been produced by a Scottish firm. Of low cost, it is specially designed to help the small farmer who cannot afford a large initial outlay for mechanical aids. The machine is operated by two men while a third feeds the sheaves into a drum. The drum, which runs at 350 r.p.m., is carried on a ball-bearing shaft. It is covered with a sheet metal and is fitted with four cast steel peg-type beaters. The pegs on the beaters run in the spaces between similar pegs on a stripper bar which can be adjusted to give suitable variations in the severity of threshing. A ribbed and knobbed concave divides grain from straw.

Although primarily intended for hand operation, provision is made for fitting on 1 to 1½ h.p. motor which would increase the speed of the drum to 600 r.p.m. (BIS).

### Imperial Institute, London, Annual Report, 1948

THE TWO MAIN SECTIONS OF THE Annual Report of the *Institute* for the year under review reflect the growing interest of the State, as well as of commerce and science in new Empire sources of minerals, abrasives, oil-seeds, plant fibres, tobacco, hides and skins, essential oils and insecticides.

The Mineral Resources Department reports a 14 per cent increase in inquiries covering a wide field. Prominent among them are questions concerning world resources of coal, bauxite and gypsum. The world coal shortage intensified the need in many regions to find suitable qualities of fuel before selecting new industrial sites. Fresh fields of bauxite are required to meet the world demand in the near future, and the information sought about new deposits of gypsum is clearly connected with developments in building research that call for the use of plaster of Paris as the principal material.

Other inquiries concern non-metallic minerals and comparatively rare and hitherto little known raw materials that are proving of such importance to modern industry. These include caesium, gallium, indium, germanium, selenium, tellurium, lithium, cerium and tantalum. Many Government departments and quasi-government organizations have also sought complete information about diatomite—its nature, occurrence, quality, working, production, transport, treatment, uses, prices, alternatives and statistics. In recent years, this mineral, mainly drawn from dollar sources, has been used in the filtration of many important food, drink, and pharmaceutical products. For some time the *Institute* has tried to find Empire substitutes, with Kenya as the chief source, and even Skye has been actively considered.

In the past few years, the *Institute* has also emphasized to Government geological surveys, private geologists and mining concerns, the possible presence of vermiculite in their respective territories. Today, with more knowledge of the nature and usefulness of the mineral, the stage during which it might be dismissed as worthless mica is passing. Private and official reports of vermiculite found in entirely new areas include Gold Coast, Nigeria, South-west Africa and Mysore, though its economic possibilities there are not yet known. South

Africa continues to be the chief Empire supply source, and new finds have been reported from northern Transvaal and Natal.

New outlets and applications for Sierra Leone micaceous haematite have been considered as a weighting ingredient in oil drilling muds, as heavy medium for coal cleaning, granules for roofing felt, welding red coatings and for use in powder metallurgy. Examination of mineral specimens from Tanganyika indicated that one of them was a member of the Euxenite-polycrase series and was "appreciably radioactive"; another was a garnet of good quality that was found suitable for abrasive work.

The other scientific section of the *Institute* is the Plant and Animal Products Department, whose staff, during the year, has prepared memoranda for consideration at numerous meetings of the Colonial Primary Products Committee. Other work of the Department includes plans for a world-wide series of analysis of the insecticides of pyrethrum flowers.

To assist the general plans for increasing world supplies of vegetable oils and fats, tests of sunflower seeds, four new and two older strains, have been made in Nigeria and Sierra Leone. In the former colony the new strains showed an oil content of 33 to 35.8 per cent and the older ones 27.6 to 28.9 per cent, though one of the latter gave a greater quantity per acre owing to a heavier plant yield. In Sierra Leone the oil content of two of the new strains was 23.6 per cent, that of two others from 27 to 30.4 per cent, while the older varieties yielded 22 to 26.5 per cent.

Tests of tobacco leaf grown in Nigeria by native farmers have shown that of 74 samples examined, 14 possessed good burning qualities and, under improved cultivation and curing, should be suitable for the continental and north African markets. Smoking trials of experimental samples from Nyasaland, where the resumed production of flue-cured leaf is under consideration, disclosed that five were promising as cigarette leaf of neutral type suitable for blending and sixth as pipe tobacco.

### Research in Lac

IMPROVING OF CROP PRODUCTION on certain host plants, selection of suitable alternative hosts of *Bracon greeni*, new moulding compositions, varnishes, paints and

enamels, fundamental work on hard and soft lac resins, hydrogenation of shellac and shellac components, effect of infra-red radiation on lac and lac fibres are some of the main features of the research work carried out at the *Indian Lac Research Institute* during the year 1947-48.

The possibilities of *Albizia lucida*, *Ficus bengalensis* and *Ougenia dalbergioides* as *Baisakhi* brood-producing hosts have been investigated. The field trials show that *A. lucida* gave the best results, the rates of brood used to brood yield being 1:4.6.

Harvesting of immature lac crop has been shown to give poor yields. Use of 60 mesh wire-gauge baskets as brood containers has been found effective in eliminating the whole range of organisms infecting lac but permits free passage of lac larvae. This practice has made possible the cultivation of disease-free lac in fresh areas and prevents wastage of stick lac.

Gammexane spray has given good results as an effective fumigant of brood lac. A mixture of ethylene dichloride (75 per cent) and carbon tetrachloride at a rate of 5 oz. per 10 cu. ft. of space has proved to be an excellent, non-inflammable fumigant.

Breeding studies of *B. greeni* on a mass scale on unnatural hosts showed that with the larva of *Tryphlepidia fructicassella*, 47.1 per cent parasitism was obtained; in the case of *E. amabilis*, 43.8 per cent parasitism was obtained.

Optimum conditions have been established for lac-urea-formaldehyde moulding powders with regard to (a) particle size, moisture content level necessary to produce blister-free samples, and (b) temperature, pressure and time of curing.

Lac-cashewnut shell liquid-sulphur compositions developed at the *Institute* show promise in plastic and varnish fields. The cashewnut shell liquid is first heated with 10-20 per cent sulphur at 200°-250°C. and the resultant mass, on combination with equal proportion of lac, yields a soft rubbery material. This material in combination with fillers like zinc oxide and asbestos gives plastic composition which could be moulded in the hot or cold.

Lac-cashewnut shell liquid varnish compositions in combination with linseed oil have been prepared. These compositions yield films which are smooth and glossy and non-tacky after only

18 hr. of drying, and have good flexibility and adhesion. Waterproof varnishes for coating paper, bullets and dummy cartridges have been formulated.

The use of lime in place of litharge as an incorporating agent in the preparation of lac-linseed oil varnishes has been further investigated and modified compositions have been developed. The products obtained by employing potassium hydroxide and potassium linoleate as incorporating agents are completely soluble in water as well as in the usual varnish solvents. A new lac-linseed oil vehicle for paints and enamels has been prepared using linseed oil (100 parts), lac (20 parts), and litharge (3 parts); from these compositions satisfactory quick-drying paints have been obtained.

Physico-chemical studies on shellac were:

Ultra-violet absorption spectra of shellac solutions; flowing birefringence of shellac solutions; X-ray structure of shellac; the dielectric properties of acetic ester of lac; the dielectric properties of lac-glycol ether; insulating properties of lac-linseed oil-lime varnish, etc.

Infra-red heating has been employed for the determination of moisture in lac. This procedure reduces the time taken for moisture determination from 24 hr. to 4 hr.

A method has been developed to isolate wax from the waste varnish residues.

### Indian Standard for Refractories

THE ENGINEERING DIVISION COUNCIL of the *ISI* has brought out a Draft Indian Standard Recommendation for Refractories for Railways. It has been prepared by an expert committee composed of representatives of manufacturers and consumers of refractories in India.

The importance of the subject can be gauged from the fact that the railways consume about Rs. 17 lakhs worth of fire-brick.

The purpose of these recommendations is to indicate the types of refractories most suited for different classes of work in the various departments and workshops of the Indian railways. The recommendations are expected to assist the railways in the standardization of designs and qualities, in issuing tenders and making purchases, and the manufacturers of refractories in arranging their production programmes.

These recommendations give the qualities of refractories for various classes of applications in different departments and workshops of the Indian railways. Some classes of work are common to workshops other than those of the railway. The recommendations may, therefore, be taken to cover similar requirements elsewhere.

Comments on the draft standard will be received till 11 September 1949, by the Director, Indian Standards Institution, Block 11, Old Secretariat, Delhi 2.

### Announcements

*Dr. M. S. Krishnan*, Director, Indian Bureau of Mines, has been appointed the Director of the Geological Survey of India, in place of Dr. W. D. West. Dr. Krishnan is the first Indian to be appointed to the post.

*Director, Coconut Research Scheme, Ceylon* — Dr. Reginald Child has relinquished the post of Director of the Coconut Research Scheme, Ceylon, which he had held since 1932. He will be succeeded by Mr. F. C. Cooke, who was Chemist (Coconut Products) with the Department of Agriculture, Malay, from 1929 to 1938, and Canning Officer of the same department from 1946 to 1949.

*Lady Tata Scholarships for 1949* — The following international and Indian scholarships have been awarded for the year 1949:

The international awards totalling £ 3,000 for research in diseases of the blood with special reference to leucaemias are made to Dr. Edith Peterson (England), Dr. M. C. Bessis (France), Dr. J. Bichel (Denmark), Dr. Pierre Cazal (France), Dr. J. Clemesen (Denmark), Dr. E. Kelemen (Hungary), Dr. Edoardo Storti (Italy), Dr. Charles Oberling (France), Dr. Jagdish Chandra Mehta (India), Pascou Atanasu (France), and Gunther Schallack (Germany).

Indian scholarships of Rs. 250 per month each for one year for scientific investigations having a bearing on the alleviation of human suffering from disease are awarded to Messrs Gangagobinda Bhattacharya (Calcutta), Bimal Kumar Sur (Mysore), K. Ramamurti (Bangalore), Gauranga Roy (Calcutta), D. V. Siva Sankar (Madras), and P. R. Srinivasan (Coonoor).

# Reports from States & Provinces

## MADHYABHARAT UNION

### Industrial Expansion

**Power Projects** — THE GOVERNMENT have sanctioned 2 large schemes for power development. The first is a thermal station to be located at Indore at a capital cost of Rs. 121 lakhs. When completed this station would supply 15,000 kW. The other scheme is a hydel scheme on the Chambal river, 200 miles north of Indore, at a cost of Rs. 9.75 crores. The reservoir will have a catchment area of 8,000 sq. miles and the project is considered to be one of the cheapest in India from the point of view of dam construction. It is expected to produce 40,000 kW. ultimately.

The Union has both long-term and short-term industrial projects; the former ones to be taken up only after the power from Chambal projects becomes available. The contemplated ones include production of rayon and electrochemicals.

The short-term projects relate to cement, power alcohol, sugar, paper, glass, ceramics and silk weaving.

**Cement** — At present, the Union produces only 60,000 tons of cement per annum and in view of the large power and irrigation projects, the demand may be nearly 100,000 tons. The Union is rich in excellent lime stone at Neemuch and it is planned to locate a cement factory of 100,000 tons capacity at this place. The capital investment on the industry is expected to be Rs. 1.25 crores.

**Power Alcohol** — There are 5 sugar factories in this region and are estimated to make available 10,000 tons of molasses for the industry. The Union's petrol requirements are in the neighbourhood of 40 lakh gal., and 5 lakh gal. of power alcohol would

be needed for admixture. It is proposed to erect a distillery with a capacity of 5 lakh gal. per year.

The starting of a sugar unit of 1,000 tons crushing capacity, caustic soda and sulphuric acid units with 5 tons capacity each and a glass factory at Guna have been approved by the Government. Sanction has also been accorded for the establishment of a pottery work at Indore. The factory will use the extensive good quality fire clay deposits near Piplia and produce stoneware pipes, acid jars, etc. The China clay deposits near Indore may be utilized for making crockery.

## WEST BENGAL

### Silver Refinery for Calcutta

AN UP-TO-DATE SILVER REFINERY is proposed to be set up in Calcutta. The plant and accessories are reported to cost Rs. 60 lakhs and the plant is expected to be ready in about 3 years.

### Institute of Jute Technology

THIS INSTITUTE, THE FIRST OF its kind in India, is being started under the auspices of the Calcutta University and will start functioning from 1950. The Institute has been founded by the *Indian Jute Mills' Association* in collaboration with Calcutta University. Capital expenditure amounting to Rs. 20 lakhs and the recurring cost of running the Institution will be borne by the *Association*.

## BOMBAY

### Research Institute for Silk & Rayon Industry

THE *Council of Scientific & Industrial Research* has received a contribution of Rs. 15 lakhs from the *Silk & Art Silk Mills' Association Limited*, Bombay, for organizing a research institute for

the industry at Bombay. The functions of the research institute will include research in weaving of silk and art silk, including rayon and dyeing and processing of silk and rayon fabrics.

The offer has been accepted in principle, subject to the approval of the Governing Body of the Council and the Standing Finance Committee of the Indian Legislature. After approval, the Government of India will make a grant to supplement the industry's contribution.

## BIHAR

### Engineering College for Sindri

A MECHANICAL AND ELECTRICAL engineering college will be set up at Sindri, in the Jharia coalfield area, close to the Sindri fertilizer plant. The Government have sanctioned a sum of Rs. 70 lakhs for this purpose. The construction work is to begin in October this year and the college is expected to start functioning in 1950.

## UNITED PROVINCES

### Cement Factory

THE GOVERNMENT WILL SHORTLY start a cement factory to produce 700 tons of cement per day at Robertsongunj, 52 miles from Mirzapur. This is the first cement factory to be state-owned and is expected to meet a fifth of the province's requirement in cement. The Government have also surveyed other localities in the province with a view to set up a few more units.

## ERRATUM

This Journal :

1949, Vol. 8, No. 7, article entitled "A Cosmic Ray Radio Sonde", page 259, L.H. col., para 1, the sentence "The results obtained will be published separately by Messrs A. S. Rao and G. S. Gokhale" should be read as "The results obtained will be published separately with Messrs A. S. Rao and G. S. Gokhale".



# INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the Gazette of India, Part II, Section 1, for June 1949.

## Plastics & Plasticizers

39014. I.C.I. LTD.: Process for fastening nylon to nylon: *Fastening of nylon to nylon by an adhesive made from a solution of nylon in a coal-tar derivative.*

39165. I.C.I. LTD.: Improved moulding compositions: *Incorporating boric acid with or without alkali earth metal oxide or hydroxide in a phenol-formaldehyde moulding composition.*

## Inorganic Chemicals

39855. DAS GUPTA: A process for the removal of hydrogen sulphide from coal gas: *Solid product obtained or recovered from flue dust contained in the blast furnace gas.*

## Organic Chemicals

40174. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ: Method for producing and refining ethyl alcohol: *The pH of the crude ethanol product is adjusted between 6 and 9 and thereafter it is fractionally distilled and hydrogenated.*

## Miscellaneous Chemicals

39339. BAYER PRODUCTS LTD., BROWN & MELLOR: Method of combating diseases of potato tubers and preparation therefor: *Using chlorinated mononitrobenzene as active substance.*

37795. INTERNATIONAL GENERAL ELECTRIC CO. OF NEW YORK LTD.: Fluorescent materials: *Mixture of calcium phosphate with a cerium compound containing 2 to 20 per cent cerium fired at 950°C. to 1100°C.*

## Food & Kindred Products

38698. DISTILLATION PRODUCTS INC.: Production of vitamin A and intermediates therefor: *Reacting corresponding unsaturated carboxylic ester with lithium aluminium hydride or lithium boron hydride.*

38785. AMERICAN CYANAMID CO.: Purification of aqueous sugar solutions: *Treating solutions with an acid and alkaline contact mass.*

## Fuels & Lubricants

40398. C. D. PATENTS LTD.: Manufacture of carbonaceous moulding compositions suitable for extrusion and of moulded carbon products therefrom: *Mixing subdivided coal with a binding material and heating the mixture at an elevated temperature.*

## Metal & Metal Products

40503. JOHN MILES & PARTNERS (LONDON) LTD.: Production of ferrochrome, high chromium steels, stainless steels and the like: *Smelting ore with alloying metal with carbonaceous*

*material in blast furnace injected with an oxygen rich fluid.*

40291. CARNEGIE-ILLINOISE STEEL CORP.: Method and apparatus for the electrolytic coating of metal strip: *Comprises passing through an electrolyte two strips forming the cathode and the anode, guiding the strips in parallel, proximate relation to each other and maintaining shields between the edges of the strips.*

38762. WILLIAM JESSOP & SONS LTD.: Alloys of an austenitic character having a cobalt-nickel-chromium bases: *Nickel chromium steel having a proportion of niobium and vanadium and also containing 30 per cent to 50 per cent.*

41076. THE MOND NICKEL CO. LTD.: Alloys: *Containing 0.75 to 2.5 per cent carbon from 1 to 6 per cent tin, from 0.03 to 0.25 per cent magnesium, from 0.5 to 4 per cent silicon, and from 0.5 to 4 per cent manganese, the balance being nickel.*

## Stone, Clays & Glass Products

39625. TURNER ASBESTOS CEMENT CO. LTD.: Improvements relating to the manufacture of reinforced asbestos cement or like sheets: *Coating asbestos cement on rotating cylinder, introducing predetermined lengths of reinforcement and cutting into shorter sheets.*

## Textile & Textile Products

40306. BOXEL-MALETRA SOCIETE INDUSTRIELLE DE PRODUITS, CHIMIQUES: Method of producing products intended for improving the quality of textile materials: *Impregnating textile with a compound obtained by condensing a carbonic acid amide with glyoxal and again condensing the product with formaldehyde.*

40428. JOSEPH BANCROFT & SONS CO.: Printing of cellulose textile fabrics: *Printing paste comprising thermosetting resin-forming materials and a vat dyestuff in free leuco base form.*

## Miscellaneous

CARDIS: Improved fibre decorticator and method of using same: *Comprising two semicircular arcs terminating in two rectangular pieces, one of the rectangular pieces having extensions at its end.*

40873. N. V. GANPULE & R. S. KULKARNI: Manufacture of primary galvanic dry cells: *Container made of plastic material.*

38612. MISTRY: An animal weight driven water pump: *Iron wheel tilting on central ball-shaped cast iron knob operates either diaphragm or plunger type pumps.*

40458. STANDARD TELEPHONES & CABLES LTD.: Pastes for printing electrical circuit elements: *Containing finely divided material dispersed in a hydrophilic liquid containing weighting agent.*

# Research Development & Utilization

**P**ROBLEMS of research development and utilization have received the expert consideration of a Committee, appointed in August 1947, by the Governing Body of the Council of Scientific & Industrial Research. The recommendations of the Reviewing Committee (*Chairman*: Sir Ardeshir Dalal), embodied in a report recently accepted by the Governing Body of the Council, are entitled to serious consideration in view of their importance to Indian industry and of their bearing on the progress of applied research in India.

The Council of Scientific & Industrial Research, since its very inception, has been seized of the importance of research utilization, and has subjected the measures in this regard to continuous review. The work of the Council in its own laboratories at Delhi and under the research schemes financed by it in the universities and research institutions all over India, have yielded, as the Committee rightly points out, "very creditable" results. No less than 104 Indian patents (with the corresponding States patents), and 8 patents in the United Kingdom and in the United States of America have been taken out by the Council, and 329 publications constituting a valuable contribution toward the advancement of learning have been issued. Many of the processes developed by the Council have brought substantial benefits, direct and indirect, to Indian industry\* and some of the patented processes have brought handsome monetary

returns to the Council. Considering the short life of the Council, and the difficulties with which it had to contend, especially during the war years, the results achieved are praiseworthy. The Committee, however, has examined afresh the possibilities of giving further impetus to research utilization so that the work of the Council may bring more direct and more substantial benefits to Indian industry.

## Present Procedure

The present procedure for dealing with the results of research may be briefly mentioned. When a laboratory investigation reaches the stage of completion and is considered ready for development and exploitation, a "non-technical note" is prepared by the research worker and passed on to the Utilization Branch. The technical aspects and economic implications, the capital required for both block and recurring expenditure, the equipment necessary, availability of raw materials, estimated costs of production and market prices, and other aspects germane to utilization are examined by the Utilization Branch, and the note, as amended, is sent to an *ad hoc* committee for scrutiny. The "non-technical note", modified and finalized by the *ad hoc* committee, is printed and circulated among chambers of commerce and other commercial bodies and industrial concerns, and the offers received for commercializing the process are considered by the Industrial Liaison Committee which examines and decides the manner in which it is to be developed and exploited.

The procedure outlined above has proved fairly satisfactory. It has been observed, however, that in several cases industrialists have abandoned processes after developing them to a certain stage without calling in the aid of the Council to improve the technique or the economics of the processes. Although

\* An instance of this was given by Sri Kasturbhai Lalbhai, a member of the Committee. He stated that "at Ahmedabad the president of the Bobbin Manufacturers' Association had informed him that millions of bobbins were being coated by the bobbin industry with *bhilawan* paint (developed by the Council) and used by the textile mills in this country. These had brought incalculable savings to the textile industry." Many more instances, equally impressive, can be cited.



the Council has declared its readiness to assist industry by providing expert technical assistance whenever required, the services of the Council have not been adequately made use of by industry. Another serious drawback is the absence of facilities for pilot-plant trials, and "processes have been given out to industry in a 'half-baked' condition with the result that many industrialists find themselves unable to bring them to a practical and profit-yielding stage". This drawback is sought to be remedied by the installation of pilot-plant laboratories in all the National Laboratories which are being set up by the Council.

### Research & Industry

The Council's efforts to provide technical aid and its attempts to release to industry only the processes whose merits have been proved by pilot-plant trials are being supplemented by measures to make Indian industry research-conscious. The effectiveness of any measure to bring research aid to industry is determined by the extent to which industry is "alive" to the benefits of science. With a few notable exceptions, Indian industry is not sufficiently research-conscious, and during the past few years, the Council of Scientific & Industrial Research has made strenuous efforts to establish co-operative research associations. These efforts are beginning to bear fruit. A research association for textiles has been established at Ahmedabad, and proposals for establishing a similar association at Coimbatore are being considered. The Tanning and Leather Manufacturers' Association has organized a research association at Madras, and the Indian Chemical Manufacturers' Association is considering the formation of a research organization on a co-operative basis. The Council also provides facilities to manufacturers to have their problems investigated under its auspices. The Vanaspati Manufacturers' Association, for instance, has taken advantage of such facilities and has placed funds at the disposal of the Council for investigations on *vanaspati*.

The Reviewing Committee has taken cognizance of these developments, and notes that "if research is to play a part in the industrial development of the country, similar to that played by research in Europe and America, much closer liaison should be established between industry and research than exists at present". The Committee

recommends that the help and co-operation of the technical staff of the Council should be made available in a greatly enhanced measure to firms entrusted with the development of the Council's processes. The reluctance on the part of firms to apply for technical assistance should be overcome, and the Council's technical staff should pay periodical visits to such firms and help them to overcome difficulties, and thereby inspire confidence in the Council's processes. The Council should be represented on all major trade associations and chambers of commerce, and the Committee recommends the appointment of a whole-time Industrial Relations Officer who should be "a practical man with business experience, capable of understanding the economic difficulties of business men and interest industry in the results of research".

### A Research Development Corporation Needed

The Committee further recommends the establishment of a National Research Development Corporation on the lines of the Research Development Corporation recently established in U.K. The objects of the U.K. Corporation, described in the *Development of Inventions Bill*, are :

- (a) To secure, where public interest so requires, the development or exploitation of inventions resulting from public research and of any other invention which, in the opinion of the Corporation, is not being developed or exploited or sufficiently developed or exploited; and
- (b) to acquire, hold, dispose of, and grant rights (whether gratuitously or for consideration) in respect of inventions resulting from public research and, where the public interest so requires, in respect of inventions resulting from other sources.

Another method considered by the Committee is that adopted by the Bureau of Agricultural and Industrial Chemistry, U.S.A., which carries out its development research either by :

- (a) demonstrating by pilot plants which show to industrialists how the process can be operated on full-production basis ; or
- (b) co-operative agreements with industry itself where a firm's plant is used to develop a process worked out on the laboratory scale by the Bureau's staff, the firm being guaranteed against any loss by the Bureau.

Pending the installations of pilot plants in the National Laboratories, the Committee recommends the adoption of procedure (b) of the Bureau of Agricultural and Industrial Chemistry, U.S.A. "The industrial policy of the Government of India", the Committee observes, "envisages the establishment of a number of important industries under State-ownership, and a large number of major industries under State control. The Research Development Corporation may take powers under the Law to secure co-operation from all the State-owned and controlled industries to develop new processes in the plant of the manufacturing firms interested in such processes on the condition that the Corporation will reimburse to the firms any loss that may be incurred."

One of the main functions of the Research Development Corporation will be "to exploit in the public interest all patents of the Council of Scientific & Industrial Research, and all such patents as may be dedicated to it not only by individual inventors, but also by universities and by institutions supported from public funds or private endowments". The Committee suggests that the proposed Corporation should make a modest beginning,

confining itself to the development of the Council's processes by utilizing the equipment available in industrial establishments. A start can be made with a capital grant of about Rs. 50 lakhs and a recurring grant of Rs. 5 lakhs, which should be increased if the activities of the Corporation justify it.

The Report of the Reviewing Committee, incorporating many practical observations and recommendations, is a document of considerable public interest. The Governing Body of the Council, at its recent meeting, accepted the Report, and with a view to implementing its main recommendation, viz. the establishment of a National Research Development Corporation, appointed a committee of experts to determine the composition and powers of the Corporation, and the terms on which it should develop and exploit scientific processes and patents. The decision to establish a National Research Development Corporation will be widely welcomed as yet another instance of the enlightened policy, consistently and steadfastly pursued by the Council of Scientific & Industrial Research, to promote and foster scientific research in the interests of industry.

# Characteristics of the Ionosphere over Calcutta (June 1949)

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(MRS.) T. K. BHAR & A. P. MITRA

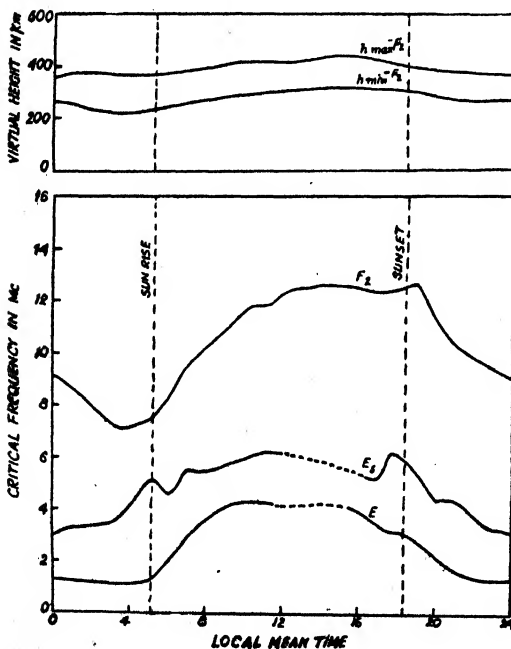
*Wireless Laboratory, University College of Science, Calcutta*

**T**HE following are the ionospheric data observed at Calcutta for the month of June 1949.

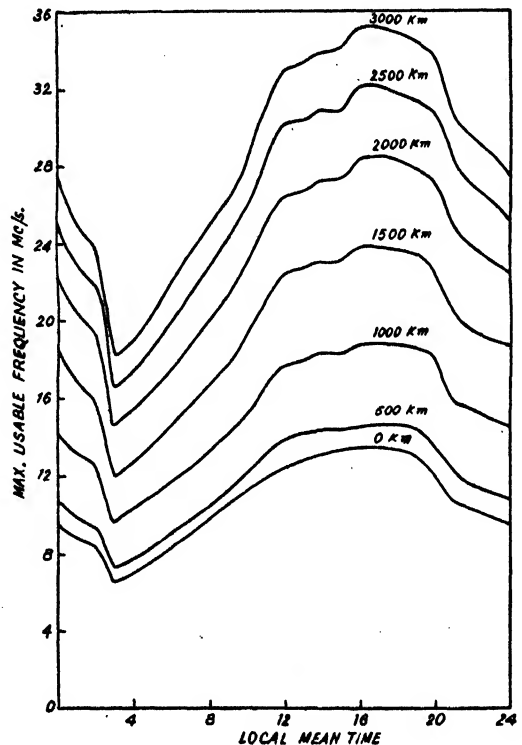
Fig. 1 represents the mean hourly values of the penetration frequencies of the  $E_s$  and  $E$  regions and the penetration frequencies and virtual heights of region  $F_2$ . The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the predictions of the maximum usable frequencies which can be used for different distances of transmission by reflection at the  $F$  region over Calcutta for the month of September 1949. Table I gives the different occasions during routine observation when sporadic  $E$  ionization was observed and the values of

the corresponding penetration frequencies and heights.

Sporadic  $E$  ionization was found to occur frequently during early morning and afternoon, often coinciding with the occurrence of local thunderstorms. High absorption of  $E$  echoes was observed during mid-day making the values of  $f^oE_s$  somewhat uncertain. The penetration frequency of region  $F_2$  was found to have an afternoon maximum and the height and thickness of the region were very high. The behaviour of this region was otherwise normal.



(5 HOURS 54 MINUTES AHEAD OF G.M.T.)  
FIG. 1 — JUNE 1949.



AT POINT OF REFLECTION.  
FIG. 2 — M.U.F. FOR TRANSMISSION VIA  $F_2$  LAYER,  
SEPTEMBER 1949.

TABLE I

MONTH & YEAR	DATE	HOURL	$f^oE_s$ Mc.	$h'E_s$ Km.
June 1949	1	00.00	2.00	90
		08.00	6.25	120
		09.00	6.00	120
		10.00	4.75	120
		11.00	6.00	135
		*12.00	6.75	135
		18.00	5.60	120
		20.00	6.00	120
		21.00	5.25	120
	2	07.00	6.45	120
		08.00	6.00	120
		09.00	6.25	120
	3	09.00	6.10	135
		19.00	3.60	120
		20.00	3.00	105
		21.00	2.60	90
		22.00	2.40	90
	4	01.00	2.00	90
	7	18.00	4.75	105
		19.00	4.00	105
		22.00	3.30	105
	8	09.00	4.75	120
		10.00	5.00	120
	9	20.00	5.20	120
		21.00	5.50	120
		22.00	4.75	105
		23.00	3.00	105
	10	00.00	4.25	105
		06.00	2.65	90
		07.00	3.50	105
		08.00	6.25	120
		09.00	5.00	120
		10.00	6.25	120
	13	18.00	4.50	120
		19.00	3.15	120
	14	08.00	4.00	105
		09.00	4.25	105
		10.00	4.50	120
	15	02.00	3.05	90
		03.00	3.15	90
		04.00	3.25	90
		05.00	3.00	90
	16	09.00	4.10	105
		10.00	4.50	105
		19.00	5.00	120
		20.00	4.75	120
		21.00	8.50	120
		22.00	6.45	120
		23.00	4.65	120
	17	02.00	2.70	105
		03.00	2.55	90
		04.00	2.35	90
		05.00	7.95	105
		06.00	7.95	105
		20.00	3.00	105
		21.00	3.50	105
		22.00	4.00	105
		23.00	3.75	105
	18	00.00	3.00	90
		06.00	3.25	90
		07.00	4.25	105
		08.00	5.00	105
		09.00	5.00	120
		10.00	5.25	120
		12.00	7.00	135
	20	23.00	3.40	105

\* Local thunderstorms.

TABLE I—contd.

MONTH & YEAR	DATE	HOURL	$f^oE_s$ Mc.	$h'E_s$ Km.
	21	00.00	3.30	90
		02.00	3.00	90
		07.00	3.70	105
		08.00	4.00	105
		19.00	4.25	105
		20.00	4.00	105
		21.00	3.75	105
		23.00	2.50	90
	22	00.00	4.25	90
		01.00	5.25	90
		02.00	5.65	90
		03.00	5.15	90
		04.00	4.10	90
		05.00	7.90	105
		06.00	3.10	105
		07.00	4.50	105
		08.00	4.70	105
		09.00	5.25	120
	23	10.00	4.25	120
		11.00	7.25	120
		17.00	5.25	135
		18.00	5.00	135
		19.00	4.85	120
		08.00	4.60	120
		09.00	4.75	120
		10.00	4.80	120
		11.00	4.80	120
	24	12.00	4.90	135
		*18.00	11.75	150
		20.00	10.50	150
		21.00	7.70	135
		22.00	4.40	120
		23.00	3.20	120
		00.00	3.00	105
		03.00	2.65	90
		04.00	5.25	105
	25	05.00	3.55	105
		06.00	4.70	105
		07.00	5.50	120
		*08.00	6.45	120
		09.00	6.50	120
		10.00	7.25	135
		11.00	7.00	135
		17.00	7.20	120
		09.00	6.70	120
	*27	10.00	6.90	135
		17.00	6.75	135
		18.00	10.40	135
		19.00	8.75	120
		20.00	4.25	120
		21.00	3.80	105
		22.00	3.00	105
		23.00	2.80	90
	28	00.00	2.70	90
		01.00	2.50	90
		02.00	2.40	90
		06.00	3.20	105
		07.00	10.50	105
		08.00	7.75	105
		09.00	8.00	120
		10.00	10.75	135
		17.00	3.25	135
		18.00	3.55	120
	29	20.00	2.70	105
		23.00	1.75	90
		00.00	2.00	90
		05.00	4.05	105
		06.00	4.90	105
		08.00	4.75	105
		17.00	4.00	120
		09.00	6.85	120
	30	17.00	4.10	120
		18.00	3.40	105
		20.00	3.25	105
		21.00	2.90	105
		22.00	4.00	105
		23.00	3.20	90

\* Local thunderstorms.

# Jellies & Related Products from Tamarind Seed Kernels

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**J**ELLIES, marmalades, etc., have so far been dietetic luxuries in our country on account of their high cost. However, with the discovery of tamarind seed jellose, it is possible to bring them within the reach of the poor man as the jellose possesses excellent jellying properties and would be far cheaper than any of the fruit pectins.

Tamarind seed jellose<sup>1</sup> forms excellent sugar jellies, with or without acids, though it differs chemically from fruit pectins, and the jellies so formed compare favourably in strength and transparency with those obtained from fruit pectins. Since the jellose is a colourless, odourless and tasteless product, its addition does not, in any way, affect the natural colour, flavour and taste of fruit juices from which jellies are to be prepared. Moreover, on account of its capacity to set even without acid, it scores over the fruit pectins. For example, it can form jellies with milk and sugar in the same way as it does with water and sugar. Another advantage is in the preparation of marmalades of particularly non-acidic fruits like figs, currants and bananas, since no acid taste need be introduced as when fruit pectins are used for the purpose. Tamarind seed jellose may also be advantageously used in the preparation of products similar to fruit preserves, wherein the form of the original fruit, either whole or cut, is to be retained.

There are indications that the jam, jelly and marmalade manufacturers are beginning to use jellose in place of fruit pectins and, therefore, it would be appropriate if a process which helps to lower the cost of production were to be worked out. It is now suggested that an aqueous extract of the kernels may be used in place of a solution of the isolated and purified jellose. The extract can be kept without any deterioration for two days at the ordinary temperature and for a much longer period in a frigidaire. The jellies formed are, however, slightly less

transparent but the reduction in the production costs far outweighs this drawback.

## Preparation of Jellies

Tamarind seed kernels (1 lb.) are crushed to small bits, and boiled with water (5 gal.) for an hour in a kettle, preferably of stainless steel, of 10 gal. capacity. The kettle may be heated directly. The contents are then discharged into a tall settling vessel, 1' in diam. and 3' in height. The next day the supernatant liquor from the settling vessel is siphoned into a mixing tank provided with a stirrer, mixed with infusorial earth (0.3 lb.) and then passed through a small filter-press, whereby a clear extract (c. 4 gal.) containing about 1 per cent of jellose is obtained. Alternatively, the liquor may be filtered through a bag filter made of heavy canvas, felt or other thick cloth, but in this case the filtrate will not be so clear. The extract is then led to a steam-jacketed, stainless-steel, open kettle of 10 gal. capacity, mixed with sugar (34 lb.) and boiled with thorough mixing. The boiling may also be done in a directly heated vessel, provided proper stirring is maintained right through the operation. If an acid taste is desired, citric acid (0.5 lb.) is added. During boiling the juice is skimmed in order to remove the coagulated material, if any. The mixture is further concentrated till its density is reduced to 65° to 70° Brix. This concentration can be conveniently judged by reference to the boiling temperature of the jelly solution, which will be 8° to 9°F. above the boiling point of water, that is 220° to 221°F. at sea level<sup>2</sup>. After the heating is over, a suitable colour and flavouring agent are added and the solution transferred into jelly glasses and allowed to set in a cool place. If intended for storage, the jelly solution, while still hot, is filled into enamel-lined jam cans, pasteurized at 180°F. for half an hour, preferably after the addition of a suitable preservative, and sealed. The yield of the jelly is about 58 lb.

Under the conditions described above for the extraction of the kernels, a liquor containing 1 per cent of the jellose is obtained. Should the concentration, however, be different (which can be estimated by precipitating the jellose from an aliquot part by alcohol and weighing it after drying<sup>a</sup>), the amounts of the sugar and the acid are adjusted for the preparation of the jelly according to the formula, jellose : acid : sugar :: 4 : 5 : 340.

#### Preparation of Marmalades

A marmalade is a jelly with thin pieces of fruit suspended in it. Though oranges and lemons are the common fruits employed, other fruits can also be used.

The fruit is shredded or finely sliced, and boiled in a steam-jacketed jelly kettle with 16 times its weight of the clarified tamarind kernel extract (1 per cent concentration). Boiling is continued till the slices become tender, water being added at intervals to make up the loss through evaporation. The actual duration of heating would naturally depend upon the nature of the fruit; it may be an hour as in the case of the hard orange peels or a few minutes as in the case of the tender banana fruit. The product is then mixed with 13.5 parts of sugar and boiled with mild stirring. Concentration is continued till the boiling point rises to 220° to 221°F. Subsequent operations are just the same as in the preparation of jellies.

#### Preparation of "Jellied Fruit"

These products are similar to fruit preserves, with the difference that jelly is used in place of syrup for the suspension of the fruit. The fruit should retain its form, either cut or whole, and should be crisp rather than soft. The essential operations are: (1) preparation of the fruit and (2) setting it in jelly.

The fruit is prepared as in the case of candied fruits by heating for a minute or two on successive days in syrups of progressively increasing concentration up to 65° Brix. and allowing it to get impregnated with sugar after each heating. The process is followed by drying. When ready, the fruit is placed in a suitable container, and the jelly solution, the preparation of which has already been described, is poured just to cover the fruit. On cooling, a firm jelly imbedding the fruit results.

The jelly-covered fruits may solve the problem of metallic containers to some extent. They are firm and can be packed in waxed paper cases enclosed in cardboard boxes.

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## Vermiculite—Its Occurrence, Origin & Uses<sup>\*</sup>

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**V**ERMICULITE, according to Dana, is a group name given to a number of micaceous minerals which are the alteration products chiefly of the micas, phlogopite and biotite. Many of them are stated to be of an indefinite chemical nature, their composition varying with that of the original mineral and with the degree of alteration. The

minerals of the group possess the peculiar property of exfoliation, slowly opening out when heated into long, worm-like threads. Jefferisite, vermiculite, culsageeite, kerrite, lennilite, hallite, philadelphite, vaalite, maconite, dudleyite, pyroselerite are some of the minerals included in this group.

The vermiculites, till recently, had remained an obscure group which found

<sup>\*</sup> Published with kind permission of the Director, Mysore Geological Department.

little or no practical application in industry. Within the last decade, however, this mineral has come to occupy a leading place among the industrially important minerals, and the production of vermiculite is on the increase everywhere. A search for the mineral is being vigorously pursued in all countries.

Last year, some of the mica-like minerals found near corundum pits in parts of the Tumkur district were tested and found to possess the characteristic property of exfoliation by expansion. A short note was contributed to *Current Science*<sup>1</sup> regarding the occurrence of this important mineral in Mysore and drawing attention of investigators to the possibility of finding better deposits in other parts of the State. Since the publication of this note considerable interest has been aroused and a concerted search for the mineral has been made. This has resulted in the location of a few important deposits where detailed prospecting has revealed the mineral to occur in quantity. The composition and distinguishing characters of this mineral, its mode of occurrence and origin, localities in Mysore State where it is found and its industrial uses are briefly described in this note.

#### Composition & Distinguishing Characters

Table I gives chemical analyses of vermiculite from (1) Bageshpura and (2) Malavanghatta.

TABLE I

	(1)	(2)
SiO <sub>2</sub>	38.10	36.06
Al <sub>2</sub> O <sub>3</sub>	17.24	20.48
Fe <sub>2</sub> O <sub>3</sub>	8.24	15.91
FeO	1.82	3.12
CaO	3.20	3.54
MgO	15.49	12.95
K <sub>2</sub> O	...	0.12
Na <sub>2</sub> O	...	...
MnO	...	Tr.
TiO <sub>2</sub>	...	0.64
H <sub>2</sub> O	17.12	7.82
	100.71	100.64

The variation in chemical composition seen is probably due to the varying degree of alteration of the original biotite and to its being intimately mixed with talc and other hydrous ferro-magnesian silicates.

The mineral is generally brown in colour with a bronzy, metallic lustre, but shades of dark-green are also common. It looks like any piece of mica but is inelastic and brittle. It is soft and has a soapy feel.

On application of sudden heat, it expands ten to fifteen times its original volume. This property serves to distinguish it from the rest of the micas. The expansion is followed by a change in colour from brown to a pleasing silvery or golden-yellow colour. The specific gravity of the unexpanded material varies from 2.5 to 2.6. This gets reduced to 0.1 on expansion. The swelling of vermiculite on heating is explained as due to the release of water between the cleavage flakes and the soapiness and oiliness as due to the presence of free alkalis on cleavage faces.

#### Mode of Occurrence & Origin

Vermiculite is generally found in the neighbourhood of altered ultrabasic rocks like peridotites and amphibolites. The presence of vermiculite on the surface is indicated by the presence of slippery soil composed of mica flakes. When closely examined, it is seen that these mica-like flakes are crowded along the borders of the ultrabasic mass in contact with intrusive bodies of igneous rock. At Bageshpura, vermiculite is found developing in an amphibolite at contact with an intrusive pegmatite. Glistening, bronze-coloured, mica-like flakes are crowded together at these contact zones giving the appearance of an altered mica schist. Where bands of amphibolite occur in the form of thin ribbons and caught-up patches in the pegmatite and gneiss, the alteration to vermiculite is complete, but where the amphibolite bands are several feet in thickness, vermiculite is seen developing only along the edges of the amphibolite mass. At Malavanghatta and at Chunchankatte, vermiculite is seen developing at the edges of a coarsely crystalline hornblende schist at contact with a muscovite-bearing pegmatite. In all these three localities, apatite occurs as grains and lumps in the vermiculite zone. It is of interest to note that apatite is found in appreciable quantities associated with vermiculite in the well-known deposits at Libby, Montana and at Palabora in Transvaal. The presence of apatite points to the hydrothermal nature of the alteration.

It is generally recognized that pegmatites intruding ultrabasic rocks get desilicated and the excess alumina in the intruding mass crystallizes as corundum at contact with the ultrabasic. The desilication has its effect upon the ultrabasic

rock too as much of the silica is absorbed. The result is that the serpentine of the ultrabasic rock gets converted into talc and phlogopite as a result of the addition of silica abstracted from the pegmatite. The large amount of water released by the transformation of serpentine to talc would probably effect the alteration of phlogopite to vermiculite. The development of vermiculite in the ultrabasic rocks at contact with corundum aplites and pegmatites is commonly noticed in all corundum areas. It is, therefore, clear that vermiculite is a contact mineral developed in ultrabasic rocks at contact of intrusive pegmatites and gneisses. Weathering by itself seems to have little action in the formation of vermiculite.

In the case of hornblende rocks, the hornblende appears to have got transformed into biotite at contact of intrusive pegmatites and gneisses. Further alteration of the biotite through hydrothermal action of water and other dissolved gases seems to have resulted in the formation of vermiculite.

#### Occurrence

*Bageshpura Area* — The country to the east and north-west of Bageshpura, a railway station on the Mysore-Arsikere line, exposes a highly cut-up ground composed of kaolinized granites and pegmatites with intercalated lenses of altered ultrabasic rocks which at places are corundum bearing. The contact zone of the ultrabasic rock with the pegmatite shows the formation of tiny flakes of bronze-coloured vermiculite. The development of vermiculite is strong only at the contacts while the interior of the ultrabasic mass remains talcose and actinolitic.

A number of bands of these ultrabasic rocks can be traced amid the gneisses in the area, showing a greater or lesser degree of development of vermiculite along contact zones. So far, no single prominent band several yards in width and hundreds of feet in length has been traced. Prospecting has shown some of the bands to pinch out at depths. The area over which the bands are distributed is very large and a careful and systematic search is likely to reveal larger and better deposits of the mineral than hitherto found. Vermiculite in this area could be collected as a by-product of kaolin mining. In one locality near the Appenhalli temple, bluish-

green crystals of apatite are seen distributed uniformly in the form of grains throughout the vermiculite zone.

*Channarayapatna Area* — Specimens collected by the late Sri P. Sampat Iyengar as biotite schist from near the apatite occurrences at Malavanghatta, a village 5 miles east of Channarayapatna, Hassan district, were found to possess the characters of vermiculite. This occurrence was taken up for detailed prospecting early this year.

The deposit of vermiculite is situated in a small water course about three furlongs NNE. of Malavanghatta. It is seen as a highly crumpled schist band on the surface and is twisted about in all directions. It is about 30' wide and can be traced for a distance of about 200'. On account of the folding, the width of the band appears to be more at a few places. The parent rock which has given rise to the vermiculite is a hornblende granulite. There are a few thin pegmatite veins but no regular outcrops. Nearby is an east-west running hornblende dyke and a band of highly actinolitic and talcose ultrabasic rock which too shows vermiculite in patches. Prospecting pits have reached a depth of 20' and show vermiculite right through to this depth. In the vermiculite zone, here and there are a few lumps of greenish apatite and also unaltered islands of bluish-green hornblende rock. Vermiculite from this area is dark and looks like biotite. In the neighbourhood there are several other minor occurrences of vermiculite in the form of thin ribbons and lenses.

*Chunchankatte Area* — Good specimens of vermiculite have been obtained from near Chunchankatte, 9 miles WNW. of Krishnarajanagara, Mysore district. The vermiculites from this area are bronze-yellow to dark-green in colour. In hand specimens, a few crystals of light-green apatite can be recognized. Vermiculite has developed in a coarsely crystalline hornblende schist at contact with a muscovite-bearing pegmatite. The vermiculite band is about 3' wide and is traceable on the surface for a distance of about 1,000'. There are several other minor bands by its side. The deposit has not yet been prospected.

*Nidavanda Area* — The highly cut-up ground to the north and north-east of Appagondanahalli, about 4 miles ENE. of Nidavanda Railway Station, shows up kaolinized gneiss with caught-up bands and shreds of black and green hornblende



rock in all stages of alteration. The borders of these schist inclusions in contact with the gneiss or pegmatite show incipient development of flakes of vermiculite. Sometimes they are in the form of minute brown flakes distributed uniformly throughout the mass, and at others in the form of large plates crowded together in narrow marginal zones. Most of the occurrences are thin and no large development was noticed. The area is extensive as at Bageshpura and further search may reveal some workable deposits.

**Pavagada Area** — It was in this area that vermiculite was first noticed. East of Nagalmadike and Bugadur, very near the State border, are a few corundum pits round which are seen a number of glistening flakes of bronze-coloured vermiculite. The locality is far in the interior and devoid of all communications and as such prospecting was not taken up.

#### World Occurrences

The earliest known deposit of vermiculite is probably the one at Libby, Montana, U.S.A., and this deposit has been described as a hill, 1,000' high, and almost a square mile in extent, wholly composed of vermiculite. Vermiculite has developed in an altered peridotite in contact with syenite and the rock is stated to contain up to 7 per cent of apatite.

The other well-known deposit of vermiculite is in the Palabora district of Transvaal, where it was first considered to be merely an accessory to apatite. Exploratory work revealed the deposit to extend over an area of nearly  $\frac{3}{4}$  mile and to a depth of nearly 80' in places. Vermiculite in this area is stated to occur in the form of platy aggregates capable of easy sorting and grading. The reserves are estimated at 1,000,000 tons.

Important Russian deposits are near Bulduin in the Ural Mountains. The deposits are stated to be of considerable extent and of commercial quality.

Vermiculite is described to occur at Bulong, east of Kalgoorli and at Yellowdin and Young river in Australia.

The world resources of vermiculite, it would appear, are limited.

#### Industrial Uses

The uses of vermiculite are based upon its property of being extremely light and at the same time somewhat refractory with low conductivity to heat and sound.

Exfoliated vermiculite weighs only 6 to 8 lb. per c. ft. as compared with sand which weighs 100 lb. and is, therefore, extensively used in concrete work in place of sand to save weight. Vermiculite concrete weighs only 25 to 50 lb. per c. ft. compared to ordinary concrete which weighs 130 to 150 lb. This effects considerable savings because of reduced columns and beam sections and of lighter foundations. The property vermiculite has of holding small pockets of air between non-conducting fibres or plates makes it a good insulator. It is, therefore, used as an aggregate in insulating plaster and concrete. Vermiculite concrete, on account of its light weight and insulating properties, is used in making panels for prefabricated houses, roof decks, etc. An inch of vermiculite plaster is stated to give 4 times the fire-proofing effect of ordinary plaster. It is reported that an insulating block of vermiculite bonded with 25 per cent of colloidal magnesium silicate withstands a temperature of 2,000°F. Vermiculite concrete bricks are used for lining furnaces to withstand temperatures up to 1,600°F. Lining domestic stoves with vermiculite concrete is stated to lead to decreased fuel consumption.

Because of the continuous channels connected with small pores which expanded vermiculite provides, it is greatly useful for sound insulation and is extensively employed in radio studios, theatres and libraries to improve acoustics and reduce noise. Monolithic cast *in situ* sets of sound-absorbing panels for aeroplane engine testing sheds have been made out of vermiculite concrete.

A new building material, "Pyrok", has been developed in Great Britain from vermiculite. It consists of vermiculite bonded by a mixture of lime and cement. It is a surfacing material which is waterproof and fire resistant and adheres strongly to any surface including wood, steel, asbestos and cement, and is free from the usual cracks evident in plaster. It is finding application in the ship-building industry as a coating to the underside of steel decking.

During the recent war, tankers that were likely to be exposed to bombing attacks had slabs and blocks of vermiculite concrete placed on their decks.

Expanded vermiculite has a very pleasing golden-yellow colour like that of gold dust and the powdered material is used in wall

paper decoration. Another use of vermiculite is as a lubricating agent. It is reported that it does not abrade and the powder has lubricating properties similar to graphite. New uses are still being discovered and with the passing of years vermiculite is tending to occupy a leading position among the industrially important minerals.

#### Production & Costs

The following information relating to the production and value of vermiculite produced in the United States of America is extracted from the *Minerals Year Book* (1946):

"Screened and cleaned vermiculite averages \$8 to \$12 per short ton. Recent information obtained from exfoliators places the loss of weight on expansion at about 5 per cent. Assuming an average price of \$75 per ton the total value of exfoliated vermiculite sold in 1946 would be approximately \$6,155,000." Table II gives figures of screened and cleaned vermiculite sold or used by producers in the United States for the years 1935-39 and 1940-46.

TABLE II

YEAR	QUANTITY, TONS	VALUE, \$
1935-39 (average)	18,486	180,297
1940	22,299	137,698
1941	23,438	125,444
1942	57,848	319,931
1943	46,645	471,595
1944	54,116	541,744
1945	64,808	648,077
1946	86,390	867,973

#### Future Scope

Considerable amount of work remains to be done in the direction of an intensive survey of large tracts of country covered by ultrabasic rocks intruded by acid and basic intrusives with a view to locating commercially important deposits of vermiculite. Laboratory experiments have to be conducted on a large scale to find out

the behaviour of local vermiculite under different temperatures. As the amount of expansion on heating is a criterion for establishing the market value of vermiculite, maximum expansion will have to be ensured by cleaning and grading the product first and subjecting it to the right temperature. A suitable plant will have to be designed for cleaning the mined product and subjecting it to the right temperature to ensure maximum exfoliation.

Again, in making use of vermiculite in concrete, experiments have to be conducted regarding the method of mixing, the proportion in which the different materials have to be mixed and similar related matters. Such concrete slabs will have to be then tested for their acoustical and sound-proofing properties. The use of vermiculite concrete in building structures has to be actually demonstrated by building experimental units. For insulation of hot pipes and as loose fill round boilers, vermiculite will find increasing use. The use of vermiculite in prefabricated houses in the form of precast slabs has to be investigated and a workable formula regarding large-scale manufacture of such slabs arrived at.

Experiments on these and other similar lines have to be carried out by our research institutions on the basis of which a flourishing vermiculite industry could be built up in this country. The world supply of the mineral appears to be short compared to the demand. A well-planned exploration of the area covered by ultrabasic rocks, it is hoped, will reveal large and better deposits than hitherto found and will make this country not only self-sufficient in respect of this wonder mineral but also permit of export outside.

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# Inter-Crystalline Failure of Bullet-proof Armour Plate

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**T**HE present work concerns armour plates made of nickel-chromium alloy steels which had developed cracks after leaving the assembly sheds. The standard procedure of subjecting the plates to specified ballistic fire tests and thorough examination for any cracks before and after assembly had strictly been adhered to and no cracking was discovered until much later. Fig. 1 shows one of the armoured carriers with a cracked plate. Except the side X, the other three sides had been welded. Side X was riveted and in the majority of armour plate failure cases, cracking started from side X. Only in one case cracking started from side Z. Cracking, as it generally occurs, is shown in Figs. 1 and 2. It will be noticed

that cracking started from the welded side A (FIG. 1) and the 3 welded sides were crack free.

Table I gives the chemical composition of the cracked plates.

Fig. 3 shows a typical crack passing through the bulge caused by bullet fire during the ballistic test. This crack had developed after the assembly of the plate into an armoured carrier and was not observed during any of the inspection stages.

## Physical Properties

The above set of plates were tested for bend, Izod impact and ballistic tests using stronger ammunition than that specified. The results are given in Table II.

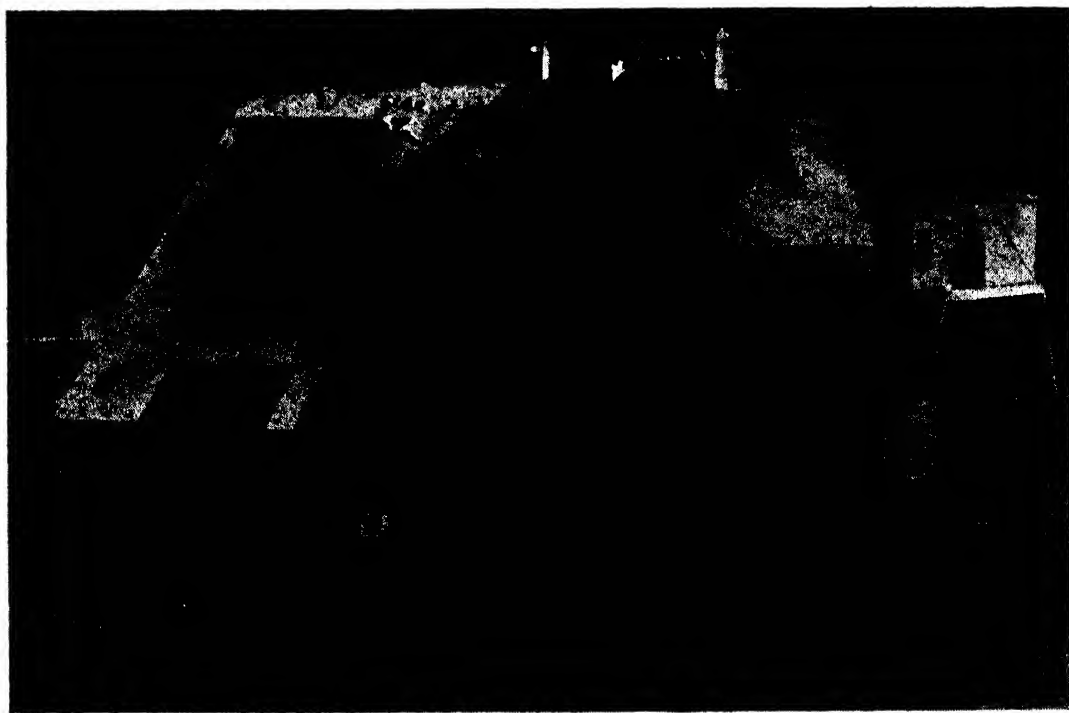


FIG. 1 — SHOWS THE CRACKED HULL OF AN ARMoured CAR ; POSITION OF CRACKS SHOWN AT A AND A<sub>1</sub>.

TABLE I—CHEMICAL COMPOSITION OF CRACKED PLATES

PLATE No.	CARBON %	MANGANESE %	SULPHUR %	PHOSPHORUS %	NICKEL %	CHROMIUM %	MOLYBDENUM %
1	0.41	0.57	0.012	0.026	4.60	1.57	0.26
2	0.40	0.56	0.011	0.032	4.33	1.53	0.44
3	0.41	0.51	0.011	0.022	0.20	1.38	0.22
4	0.40	0.56	0.010	0.036	4.39	1.43	0.31
5	0.39	0.54	0.010	0.036	4.38	1.46	0.34
6	0.37	0.48	...	...	4.33	1.39	0.43

TABLE II—PHYSICAL PROPERTIES OF CRACKED PLATES

PLATE No.	ACTUAL THICKNESS, in.	BEND TEST ANGLE OF BEND BEFORE FRACTURE deg.	IZOD IMPACT TEST, ft./lb.	FIRING TESTS	
				With 12 mm. ammunition (as specified)	With 14 mm. ammunition
1	0.487	...	...	Test O.K.; no cracking from bullet mark	O.K.; no cracking
2	0.492 0.502 0.505	68 ...	6, 5, 6 ...	Test O.K.; slight cracking at bullet mark and near a Brinell impression developed in storage	Slight cracking on the other side
3	0.484	...	...	Test O.K.; slight cracking developed at bullet mark	Slight cracking on the other side
4	0.487 0.495 0.490	35 ... 75	... 5, 6, 5	Test O.K.; slight concentric cracking at the bullet mark on the other side	Bulging and slight cracking on the other side
5	...	...	...	Test O.K.; slight cracking at the bullet mark on the other side during storage	No cracking at the bullet mark
6	0.492 0.497	... 53	11, 12, 10	Slight cracking developed at the bullet mark	Cracking on the other side with slight bulging

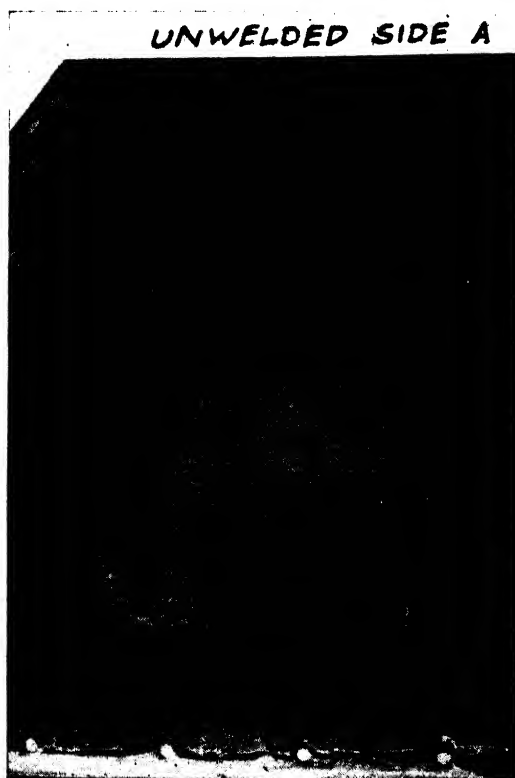


FIG. 2—SHOWS THE STARTING OF THE CRACK FROM THE UNWELDED SIDE OF THE ARMOUR PLATE.

It was considered necessary to examine along with the cracked plates some more plates of 12 mm. thickness which had withstood the ballistic tests and had developed no cracks so far. Tables III and IV represent the properties of a typical uncracked plate.

It will be observed that the cracked plates exhibit almost the same physical properties as the sound plate No. 7.

#### Surface Hardness of Plates

It can be gathered from Figs. 4-7 that the surface hardness of plate No. 1 was fairly



FIG. 3—SHOWS CRACKS PASSING THROUGH A BULGE BY BULLET FIRE; A; CRACK; B; BULLET MARK.





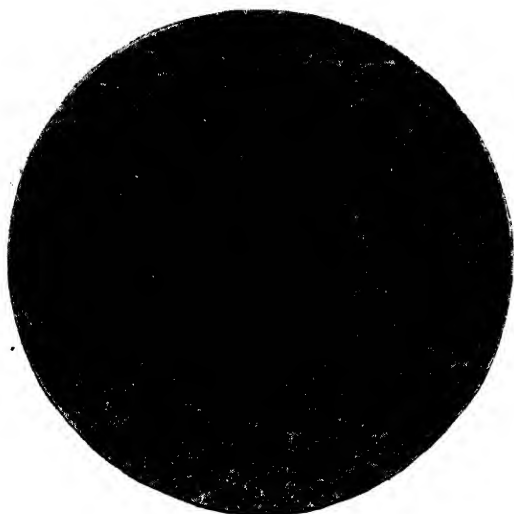


FIG. 8 — NITAL ETCH SHOWING THE MICRO-STRUCTURE OF THE ARMOUR PLATE.  $\times 100$ .

micro-banding as shown in Fig. 10. The banding is believed to be due to heterogeneity and lack of opportunity for interdendritic diffusion in the ternary Ni-Cr-Mo alloy steels. A transverse section through one of the segregated zones known as "ghosts" is illustrated in Fig. 11. The existence of such abnormalities within the mass is a source of weakness.

#### Types of Cracking\*

The following represent the different types of cracking classified on the basis of their origin :

1. *Gas-cut Cracking* — This develops as a result of gas-cutting a plate which, owing to imperfect or lack of annealing treatment, possesses considerable hardness. The cracks originate at various points on the edge and progress inwards in a straight line, traversing the whole section of the plate. Fig. 12 shows the surface of such a gas-cut crack which is generally about 1" to 1.5" in length. The micro-structure is substantially altered at the edge of the gas-cut crack. Gas-cutting cracks are mostly trans-crystalline and are due to sudden cooling from high temperature; these are not caused by the gas.

2. *Hair-line Cracks* — Numerous investigations regarding the origin, nature and prevention of hair-line cracks in nickel-chromium steel have been conducted by several workers. It has been observed that hair-line cracks are formed within the mass of the material during the cooling of steel,

and the rate at which the steel passes through the 200°C. region is the determining factor.

It is also suggested that formation of hair-line cracks does not occur immediately with the production of the material but follows storage at room temperatures. This apparent induction period is explained as due to the slow rate of diffusion of hydrogen in steel.

In the present investigation, hair-line cracking was observed in plate No. 3, the cracks running within the section of the plate  $\frac{1}{4}$ "-1" away from the edge. Under high magnification, it was observed that these cracks were inter-crystalline in nature as shown in Fig. 13. Other examples of hair-line cracking met with in this work are shown in Figs. 9 and 14.

*Cold Work Cracking (Brinell & Bullet-cracking)* — A piece was cut in plate No. 3 on whose surface, next to the cut edge, a Brinell hardness test was taken during routine testing. The piece was given a microscopic polish on the sides and examined for hair-line cracks. On the side of the Brinell impression, a fine crack was noticed to start from the latter's impression and proceed downwards. This is shown in Fig. 15. The crack was observed to be inter-crystalline and exhibited no alteration of micro-structure in its vicinity. The hardness in this case was 460-467 Brinell.



FIG. 9 — NITAL ETCH, SHOWING THE SORBITE STRUCTURE AND INTER-CRYSTALLINE CRACKING OF ARMOUR PLATE.  $\times 300$ .



FIG. 10 — CUPRIC ETCH, SHOWING MICRO-BANDING IN ARMOUR PLATE.  $\times 300$ .



FIG. 11 — TRANSVERSE SECTION THROUGH "GHOST" IN ARMOUR PLATE.  $\times 100$ ; A: MICRO-BANDING; B: HIGH NICKEL AREAS; C: LOW NICKEL AREAS.

The type of cracking described above is designated "Brinell-cracking".

Another piece from the same plate was given a Brinell test and etched in 50 per cent aqueous solution of hydrochloric acid at a temperature of  $70^{\circ}\text{C}$ . for  $\frac{1}{2}$  hr. The Brinell cracking which developed thereby is shown in Fig. 15. Similarly, the surface of the piece developed Brinell-cracking after hot hydro-

chloric acid etching. It is possible that during the hot-etching treatment the crack further opened out or cracks may actually form during the hot-etching treatment itself owing to the release of the strain in the material in the vicinity of a Brinell impression. This occurs only when the hardness is over 400 Brinell; below this range hot-etching treatment does not give rise to Brinell-cracking. Brinell-cracking is, therefore, the cracking that develops instantaneously or during storage after Brinelling, and without the aid of hydrochloric acid hot-etching treatment.

In a few cases, Brinell-cracking developed during storage, i.e. 10 to 12 hr. after Brinelling, but generally it is instantaneous. As will be shown later, Brinell-cracking results only when the hardness of the steel is above a certain range. Below that range no instantaneous or storage cracking develops after Brinelling. Examples of Brinell-cracking are shown in Fig. 15.

Similarly "bullet-cracking", namely cracking which develops in the section of the material instantaneously or some time after the bullet impact, was also observed. For this cracking, a certain hardness range had to be exceeded. In all such cases of bullet-cracking, the material had passed the ballistic fire test, having withstood penetration, flaking or surface radial-cracking at the bullet impression on both sides of the plate. An example of the above bullet-cracking in plate No. 7 is shown in Fig. 16. It is evident that bullet-cracking opens out considerably on hot hydrochloric acid etching treatment. In another case (high nickel plate, 4.5 per cent Ni.), a deep surface crack passing through the bullet hole developed in storage. This might have been initiated from internal bullet-cracking opening out at the surface eventually. Fig. 16 shows this type of storage-cracking.

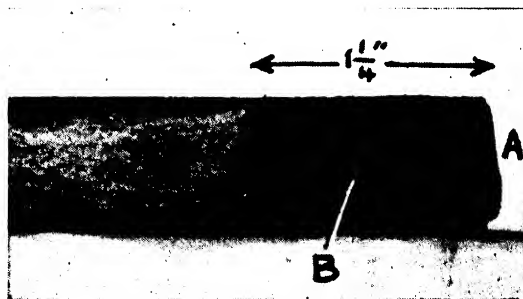


FIG. 12 — GAS-CUT CRACK IN ARMOUR PLATE; A: EDGE OF THE PLATE; B: GAS-CUT CRACK.





FIG. 13 — NITAL ETCH SHOWING INTER-CRYSTALLINE CRACKING IN BULLET-PROOF ARMOUR PLATE.  $\times 300$ .

All the above test plates had received a preliminary heat treatment. They were first annealed at  $630^{\circ}\text{C}$ . after rolling, then normalized from  $840^{\circ}\text{C}$ . and air hardened. This was followed by tempering at  $400^{\circ}$ - $425^{\circ}\text{C}$ . and air cooling.

It was observed that a plate which had received the above treatment with a resultant hardness of over 430-450 Brinell may be liable to "Brinell-cracking". It is difficult to define a strict range of Brinell-hardness with regard to Brinell-cracking. A plate with a hardness of less than 410-420 Brinell was observed to be immune to Brinell-cracking. Those plates, which were susceptible to Brinell-cracking, were equally prone to bullet-cracking.

Experiments were undertaken to study the effects of annealing and furnace cooling from  $860^{\circ}\text{C}$ . in relieving stresses in the material following normalizing at  $860^{\circ}\text{C}$ . and tempering at  $425^{\circ}\text{C}$ . by air-cooling. It was shown that annealing at  $860^{\circ}\text{C}$ . did not impart immunity to Brinell-cracking which, as before, depended upon the final hardness of the material acquired after subsequent normalizing and tempering treatments. The  $860^{\circ}\text{C}$ . annealing treatment did not soften the hard, cracked plate so much as the sub-critical  $630^{\circ}\text{C}$ . annealing treatment did, which shows the hardening effects of alloying elements like nickel, chromium and molybdenum even with rates of cooling so slow as the furnace cooling. Typical experimental results are given below.

Pieces annealed at  $860^{\circ}\text{C}$ . (TABLE V) were normalized and tempered and tested with the following results:

PLATE No.	BRINELL HARDNESS BEFORE ANNEALING	BRINELL HARDNESS AFTER ANNEALING AT $860^{\circ}\text{C}$ .	BRINELL HARDNESS AFTER ANNEALING AT $630^{\circ}\text{C}$ .
2	444; showed Brinell-cracking	429; absence of Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)	311; no Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)
3	488; showed Brinell-cracking	409; absence of Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)	293; no Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)
6	467; showed Brinell-cracking	388; absence of Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)	293; no Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)
7	454; showed Brinell-cracking	410; absence of Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)	280; no Brinell-cracking ( $1\frac{1}{2}$ hr. soaking)

The piece from plate No. 3 which had been tempered at  $425^{\circ}\text{C}$ . (TABLE VI) was heat treated as follows:

PLATE No.	BRINELL HARDNESS AFTER ANNEALING AT $860^{\circ}\text{C}$ .	BRINELL HARDNESS AFTER NORMALIZING FROM $860^{\circ}\text{C}$ .	BRINELL HARDNESS AFTER TEMPERING AT $425^{\circ}\text{C}$ .	REMARKS
2	429	495	406	Brinell - cracking was noted after normalizing from $860^{\circ}\text{C}$ . No Brinell-cracking after tempering at $425^{\circ}\text{C}$ .
3	409	488	415	do
6	388	488	415	do
7	406	488	415	do



FIG. 14 — NITAL ETCH SHOWING INTER-CRYSTALLINE FAILURE OF ARMOUR PLATE.  $\times 300$ .



FIG. 15 — BRINELL-CRACKING ; A : STORAGE CRACKING ; B : CRACK FREE ; C : MICRO-BANDING.

The piece from plate No. 3 which had been annealed at 630°C. (TABLE V) was heat treated as follows.

These two renormalized pieces (TABLES VII and VIII) were again tempered at 425°C. with the following results (TABLE IX) :

TABLE VII

PLATE No.	BRINELL HARDNESS AFTER TEMPERING AT 425°C.	BRINELL HARDNESS AFTER RENORMALIZING FROM 860°C.	REMARKS
3	415	495	Again exhibited Brinell-cracking after renormalizing

TABLE VIII

PLATE No.	BRINELL HARDNESS AFTER ANNEALING AT 630°C.	BRINELL HARDNESS AFTER RE-NORMALIZING FROM 860°C.	REMARKS
3	293 (no Brinell-cracking)	492	Exhibited Brinell-cracking after normalizing

TABLE IX

PLATE No.	BRINELL HARDNESS OF PIECE (TABLE VIII) AFTER NORMALIZING (a) FROM 860°C.	BRINELL HARDNESS OF PIECE (TABLE VII) AFTER NORMALIZING (b) FROM 860°C.	BRINELL HARDNESS AFTER TEMPERING AT 425°C. (a) (b)	REMARKS
	492	495	415 415	Exhibited no Brinell-cracking after tempering in both cases, (a) & (b)

The above experiments show that Brinell-cracking is independent of the mode of pre-annealing treatment of the piece, and demonstrate its relation to the resultant final Brinell hardness of the material.

#### Temper Brittleness

It has been recorded that nickel-chrome steels, when tempered between 250°-450°C. and air cooled, show minimum values in respect of Izod impact toughness. This phenomenon is termed temper-brittleness. Below and above this temperature range the Izod impact toughness exhibits a consistent rise. Even when a higher tempering temperature like 550°C. is employed, low impact values are obtained if the steel is allowed to cool slowly after tempering, as in air cooling ; but if it is quenched in oil or water from the higher tempering temperature, better impact values are obtained. The mode of cooling after tempering from temperature within the "temper-brittleness" range or from below it does not materially affect the impact toughness value. The slower the post-tempering cooling from higher temperatures, the lower is the final impact value and vice versa. The hardness of the differently post-tempered, cooled pieces will be practically the same.

As has been shown earlier, the plates under investigation possess Izod impact values of 5 to 10 ft./lb.

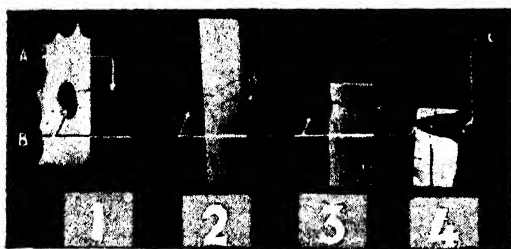


FIG. 16 — BULLET-CRACKING; A: CRACK DEVELOPED DURING STORAGE; B: BULLET HOLES; C: BULLET-CRACKING.

It was thought necessary to investigate whether an improvement in the impact values of plates could be effected by varying the tempering temperature above the temper-brittleness temperature range and below it and by varying the mode of cooling thereafter, without materially affecting, as far as possible, the degree of hardness of the material.

A piece from cracked plate No. 3, which had received the annealing treatment shown in Table VI, was renormalized from 860°C., after which it attained a hardness of 500 Brinell. It was then tempered at 550°C. for 1½ hr. and cooled in an air-blast. It showed an Izod impact value of 19.36 and 20 ft./lb., i.e. an average of 19.6 ft./lb. and a hardness of 370 Brinell. The final hardness was on the low side. This heat-treatment was termed X 1.<sup>a</sup>

Another piece of plate No. 3 which had received the heat-treatment indicated in Table V was renormalized from 860°C. to give a hardness of 492 Brinell. It was then tempered at 550°C. for 1½ hr. and oil-quenched. It gave an Izod impact value of 32, 24 and 28 ft./lb., i.e. an average of 28 ft./lb. The final hardness was 375 Brinell, again on the low side. This heat-treatment was termed X 2.

The treatments X 1 and X 2 showed that pre-annealing treatment at 860°C. or 630°C. gives practically the same final hardness after normalizing from 860°C.

It was regarded necessary to repeat these tempering experiments with a view to get a higher final hardness.

Two specimens were obtained from the piece which had received the heat-treatment X 1. Both were renormalized from 860°C. thereby hardening them to 495 Brinell. Both were tempered at 500°C. for ½ hr. but one was cooled in air-blast and the other oil-quenched from the tempering temperature.

The former gave an Izod impact value of 11 and 15 ft./lb. and hardness of 420 Brinell and did not exhibit Brinell-cracking. The latter possessed an Izod impact value of 14 and 20 ft./lb., and hardness of 410 Brinell and was also free from Brinell-cracking.

The piece from heat-treatment X 2 was similarly hardened from 860°C. and tempered at 500°C. followed by water quenching. This gave a hardness of 430 Brinell and an average impact value of 20 ft./lb. and was immune to "Brinell-cracking". The hardness of 430 Brinell and Izod impact value of 20 ft./lb. are in contrast to 5 to 6 ft./lb. Izod impact value previously obtained (TABLE II).

It was thought that by proper normalizing at 860°C. and tempering at 500°C. and by suitable adjustment of their operational details, such as soaking period and the subsequent rate of cooling, a final hardness of 450 Brinell or over and an improved Izod impact toughness could be conferred together with immunity to Brinell-cracking of the material, which is susceptible to Brinell-cracking after tempering at 400°-425°C.

By tempering above the temper-brittleness range, i.e. above 500°C., there is a risk of the softening of the plate to 400-450 Brinell or below; a hardness of 450 Brinell or over is considered necessary to withstand over-matching with angle fire.

It is, however, plausible to suggest that a plate tempered at a higher temperature (500°C.) with its improved beneficial impact toughness of 20 ft./lb. average, and possessing a hardness between 400-450 Brinell, may be equally good if not better, in being able to withstand over-matching with angle fire than the present material with its poorer Izod impact toughness and a hardness of over 450 Brinell.

Some experiments were performed on pieces from plate No. 7, which were renormalized from 860°C. to harden them to 495 Brinell. These were then tempered at 400°C. within the temper-brittleness temperature range, followed by water quenching in one case and by oil quenching in the other. The former possessed 420 Brinell hardness and showed an average Izod impact value of 8 ft./lb., the latter gave 430 Brinell hardness and an average Izod value of 9 ft./lb. Both possessed immunity to Brinell-cracking. Their Izod impact values were about the same as in the original condition.

Specimens from plate No. 3, treated as shown in Table VI with a hardness of 415 Brinell, were renormalized at 860°C., tempered at about 220°C., followed by oil quenching in one case and water quenching in the other. The following are the results.

Another piece from the original cracked plate No. 3 was treated as follows.

storage or in service, owing to the dissipation and relief of internal stresses introduced into the material during various mechanical and heat treatments.

### Conclusions

The hardness of the armour plates tested varied from 460 to over 550 Brinell, and the

TABLE X

PLATE No.	INITIAL HARDNESS (BRINELL)	HARDNESS AFTER RENORMALIZING FROM 860°C., BRINELL	TEMPERING TREATMENT AT 200° TO 220°C.; MODE OF COOLING AFTER TEMPERING	HARDNESS AFTER TEMPERING, BRINELL	REMARKS REGARDING BRINELL-CRACKING
3	415	499	Oil-quenched	495	No tendency to Brinell-cracking
3	415	499	Water-quenched	495	do

TABLE XI

PLATE No.	INITIAL BRINELL HARDNESS	HARDNESS AFTER RENORMALIZING FROM 860°C., BRINELL	MODE OF COOLING AFTER TEMPERING AT 220°C.	HARDNESS AFTER TEMPERING, BRINELL	REMARKS REGARDING BRINELL-CRACKING
3	477	503	Air-blast cooling	499	After tempering showed immunity to Brinell-cracking

It can thus be inferred that by tempering at low temperature, a high degree of hardness, essential to withstand over-matching with angle fire, can be conferred on the plate.

"Brinell-cracking" actually implies the rupture of material with a high degree of hardness under cold-working loads such as are involved in Brinelling, bullet fire, hammering for flattening the plate, shock of riveting, fitting up and assembly stresses, etc. It is easily understood that such minute rupture cracks appear at the edges of the plate where cooling occurs more rapidly than in the interior after normalizing and which are liable to be harder than the rest of the plate. It is further evident that these minute cracks, once formed, are liable to open out in course of time, in

hardness across various sections of the plates exhibited wide variations.

To prevent cracking of these plates it may be necessary to keep the hardness near about 420-435 Brinell. In doing so, however, resistance to over-matching with angle fire may have to be sacrificed.

If the tempering is carried out at 500°C., the final hardness falls down to 410-400 Brinell or lower, but the impact toughness will greatly improve.

The utmost care and uniformity of operation should be ensured during the heat-treatment so that a plate is uniformly hard all over and has minimum cambering effects. The hammering operation for straightening and flattening the plate should be limited, or be performed skillfully to prevent excessive internal stresses.

# REVIEWS

**Trace Elements in Food**, by G. W. Monier Williams (Chapman & Hall Ltd., London), 1949, pp. 511. Price 30s.

THE BOOK DEALS WITH SUCH ELEMENTS AS are likely to be present in foods either naturally or adventitiously. It is surprising to note the number of elements which are inadvertently introduced in foods and the numerous ways in which they find such entrance. The use of (a) insecticides and fungicides in spraying plants, grains, seeds or fruit; (b) preservatives in canning fruits and vegetables; (c) chemicals in the preparation of some foods and beverages are only a few of the important sources of some toxic and other comparatively innocuous elements appearing in food. Considerable research in food technology has minimized the chance of such elements being found in undesirable amounts but it is fairly clear that the possibility of contamination is not altogether removed. A constant watch on the use of newly developed methods of processing foods is, therefore, absolutely essential. Each of the elements included in the book has been dealt with in regard to (a) its occurrence in food or possible sources of contamination when the element is not naturally found in foods; (b) its importance in plant and animal life; (c) the average daily intake by human beings; (d) toxic effects, if any, of excess intake when it occurs; (e) absorption and metabolism, and last but not the least; (f) methods of estimation in biological materials.

Under each of the above headings the treatment of the subject is adequate and the arrangement of matter conveniently disposed. The author's previous experience in food research has provided the necessary background enabling him to do full justice to this important subject. The analytical portion of each chapter especially is of great value since it presents a critical review of the methods most suitable for analysis of trace elements in foods. There was an urgent need of a book of this type in English in which all the relevant information on the occurrence, physiology, toxic effects and methods of analysis of trace elements in foods was presented for ready reference. Those interested in food research must feel

grateful to Dr. Monier Williams for making good that need in so capable a manner. There is ample reference material given at the end of each chapter and a suitable index is appended at the end.

V. N. P.

**The Periodic Partial Failures of American Cottons; Their Causes & Remedies, Scientific Monograph No. 2** (revised second edition), by R. H. Dastur (Indian Central Cotton Committee, Bombay), pp. ix+172. Price Rs. 6.

IN THIS REVISED SECOND EDITION OF THE monograph by Prof. R. H. Dastur, the original title has been modified by the omission of the words "in the Punjab" as it includes 2 additional chapters on the investigations conducted in Sind. As the first edition of this monograph was reviewed in this *Journal*, reference will be made only to the work carried out in Sind.

Since their introduction into large-scale cultivation in Sind, after the completion of Lloyd Barrage in 1932, the Sind-American cottons have also been reported to suffer from bad opening of bolls, although damage on the same extensive scale, as in the Punjab, rarely occurred. The same causes and remedial measures, as in the Punjab, appeared to hold good for the "bad opening" of bolls in Sind. The climate of the southern and eastern tracts which constitute the major area under American cottons in Sind is, however, comparatively mild and to this is attributed the absence of extensive damage due to "bad opening".

Prof. Dastur has also investigated the conditions associated with red leaf disease. He thinks that there may be several different causes of red leaf disease as the leaves of *hirsutum* cottons have a tendency to redden whenever they become senescent either prematurely or at the end of the life cycle. The red pigment also develops in the leaves of *hirsutum* cottons as a result of injury to, and subsequent death of cells caused by, *jassids*. Prof. Dastur's investigation deals, however, with the red leaf occurring in Sind in absence of *jassid* injury. Though the yellow red leaf was associated with light

sandy soils, both in Sind and in the Punjab, Prof. Dastur attributes its frequent and widespread occurrence in south and east Sind to high night temperatures during the fruiting period which hastens the maturation of the crop. Under these conditions bolls opened in 32 to 40 days after setting as against 45 to 70 days required in the middle Sind and the Punjab and thus the rapid maturation of the crop rapidly depleted the leaves of nitrogen and they turned yellow and subsequently red, particularly in light sandy soils (over 60 per cent sand) deficient in nitrogen. In another type of leaf-reddening the green colour changed to red without the intervening yellow and it occurred in comparatively heavy soil. Manuring with nitrogen did not ameliorate this condition as it did in the case of "yellow red". It is unfortunate that further work in this direction could not be continued.

To conclude in the words of Sir John Russell: "The monograph represents the results of years of patient scientific work honestly and competently carried out, and it cannot fail to help the growers and to stimulate scientific inquiry in connection with other crops also."

R. D. ASANA

**Growth Substances & Their Practical Importance in Horticulture**, by H. L. Pearse, Technical Communication No. 20 (Commonwealth Bureau of Horticulture & Plantation Crops), 1948, pp. 233. Price 12s. 6d.

IN THIS PUBLICATION DR. PEARSE HAS PRESENTED a balanced review of widely dispersed literature on the application of synthetic growth substances in the propagation of plants, in improving seed and producing seedless fruit, in preventing pre-harvest drop and as selective herbicides. The application of other effects of growth substances, viz. on bud inhibition, flowering in pine-apple, healing of wounds, abscission and on ripening of fruit in storage is also discussed. Other useful features of the book are an exhaustive index of results obtained with cuttings of various species (on rooting) and a list of references.

Students and research workers owe a debt of gratitude to Dr. Pearse for the pains taken by him in compiling an exhaustive source of information on this fast-developing branch of applied plant physiology.

R. D. ASANA

**Ultrasonics**, by Benson Carlin (McGraw-Hill Book Company), 1949, pp. xi+270. Price \$5.00.

THE BOOK UNDER REVIEW DEALS WITH THE engineering aspects and application of ultrasonics. The subject dealt with in this book is covered in 10 chapters as under.

Chapter I and II deal with ultrasonic waves, including ultrasonic propagation, refraction and generation. Different modes of cuts of quartz crystal used in the generation of ultrasonic waves are described in a general way in chapter III. Description of crystal holders for ultrasonic use follows in the next chapter. After a general description of resonance and reflection phenomena in chapter V, chapter VI continues with continuous wave ultrasonic systems. Chapter VII gives an account of pulsed ultrasonic systems. Dispersion and coagulation, degassing, some of the chemical effects and applications in metallurgy are enumerated in the chapter following (VIII). Elementary description of magnetostriction is given in chapter IX. The book closes with a chapter on practical considerations in the application of ultrasonics, such as testing raw materials, finished materials, measurement of thickness, its use in television, signalling, medicine, etc.

While the book cannot claim to cover the important fundamental researches made in the last 15-20 years, especially on the optics of ultrasonics, yet it gives, in a sketchy way, a description of a few of these results. The book is very weak on the theoretical treatment of the subject as a whole, but it is very comprehensive in the description of minor details, not readily found in other books, which are nevertheless very useful for the experimenter; as for example, whole chapters have been devoted to the description of crystals and crystal holders for use in ultrasonic investigation, while useful hints on crystal plating, crystal cleaning, etc., are also to be found in this volume.

On the whole, one would find that this book rather supplements the well-known treatises on the subject by Bergmann and by Heidemann; and, though the treatment of the subject is elementary, the book is an useful addition and can be read with advantage, particularly by the beginner.

S. PARTHASARATHY

**Introductory Notes on Chromatography**, compiled and published by the British Drug Houses, Ltd., 1948, pp. 40.

THIS BOOKLET PROVIDES A CONVENIENT means of introducing the subject of chromatography to those who have not hitherto given it any particular attention. While the treatment in general has necessarily been cursory, some of the applications, viz. separation and purification of vitamins, alkaloids, vegetable and mineral oils, and amino acids, have been described with more detail than one might expect to find in a monograph of this nature.

Part I of the monograph is devoted to general principles of chromatography. The subject-matter has been briefly but lucidly dealt with. Apparatus employed and procedural details are mentioned briefly. Adsorbents in particular have been very well described and the material under this head is useful in the selection of proper adsorbents. These adsorbents are readily available from the *B.D.H. Ltd.* Solvents and eluants are included in this part.

Part II deals with applications of chromatography under the following heads: vitamins, pharmaceuticals, carotenoids, vegetable and mineral oils, amino acids, miscellaneous organic compounds and inorganic ions. The last item under this head is of particular interest. What has been described in this part gives a very good idea of the utility and future possibilities of the principle of chromatography.

The booklet, with useful references and a list of standard works on the subject, is a handy and useful publication. Research workers may obtain copies of this booklet free of charge on application to *British Drug*

*Houses (India) Ltd.*, Ballard Estate, Post Box No. 1341, Bombay 1.

A. K.

**Isotopes & Their Application in the Field of Industrial Materials**, by Dr. Paul C. Aebersold (A.S.T.M. Headquarters, Philadelphia), 1948, pp. 28. Price \$1.

THE 1948 A.S.T.M. EDGAR MARBURG Lecture by Dr. Paul C. Aebersold, Chief of the Isotopes Division, Atomic Energy Commission, has now been published in the form of a 28-page booklet.

The author, after some general comments pointing to the significance and inter-relation of atomic energy and industrial materials, where he states, among other things, that science and industry are mutually dependent for progress and support and are stimulating to each other, discusses general research dividends, useful atomic power, induction of chemical and physical effects, and finally in some detail the applications of radioactive and stable isotopes. He points out that radio-isotopes of most of the elements can now be made in the atomic pile and that production at Oak Ridge is sufficient to meet American domestic needs with some over.

The author covers isotope properties, production, and measurement, with pertinent references to facilities and safety precautions. There is, also, a section devoted to the basis for isotope applications. Quite a portion of the lecture covers the use of these materials as tools for analysis with numerous specific applications. There is a discussion on stable isotopes and the lecture closes with general conclusions.

This 28-page booklet, in heavy paper cover, can be procured from A.S.T.M. Headquarters, 1916 Race St., Philadelphia 3, Pa.

A. K.

# Evaluation of Paint Films

S. M. DAS GUPTA & K. K. PURI

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**T**HE final rating of a paint film from a study of its behaviour under various weathering conditions such as light, humidity, and temperature changes is an important problem for the paint industry. In this paper, a method is reviewed which may be of value in correlating different types of failures.

The worth of a paint film is judged by its protective and decorative power. The protective power is evaluated from the ratings of certain defects or failures which are due to the weathering action of surrounding atmosphere. They are: checking, cracking, flaking, or peeling, erosion, scaling, mildew attack and chalking. The decorative power, on the other hand, is evaluated by colour, gloss, dirt accumulation and chalking. The study of the durability of a paint film has hitherto been made from a qualitative point of view, and durability has been expressed in vague terms, e.g. good, fair, bad, etc. It is not, therefore, possible to understand or reproduce the results

obtained by other workers. Holley<sup>1</sup> and Brown<sup>2</sup> have pointed out the diversity and lack of uniformity in reporting outside exposure tests. Some paint technologists denote, by arbitrary figures, the different types of failures. Although long experience in the field is of some value in such reporting, their reproducibility and intrinsic value are doubtful. Paint technologists, therefore, require a reliable and reproducible quantitative method of expressing exposure test results. Typical paint failures being many, a method of expressing durability by one quantitative figure would be invaluable.

Many attempts have been made by various workers for evolving a satisfactory method but without success. The *Federation of Paint & Varnish Production Clubs*<sup>3</sup> (U.S.A.) has evolved a method for expressing paint failures on a quantitative basis. After careful scrutiny, paint failures have been classified into 6 groups and indicated by 0, 2, 4, 6, 8, 10, according to the degree of failure; 10 indicating no failure and 0 indicating

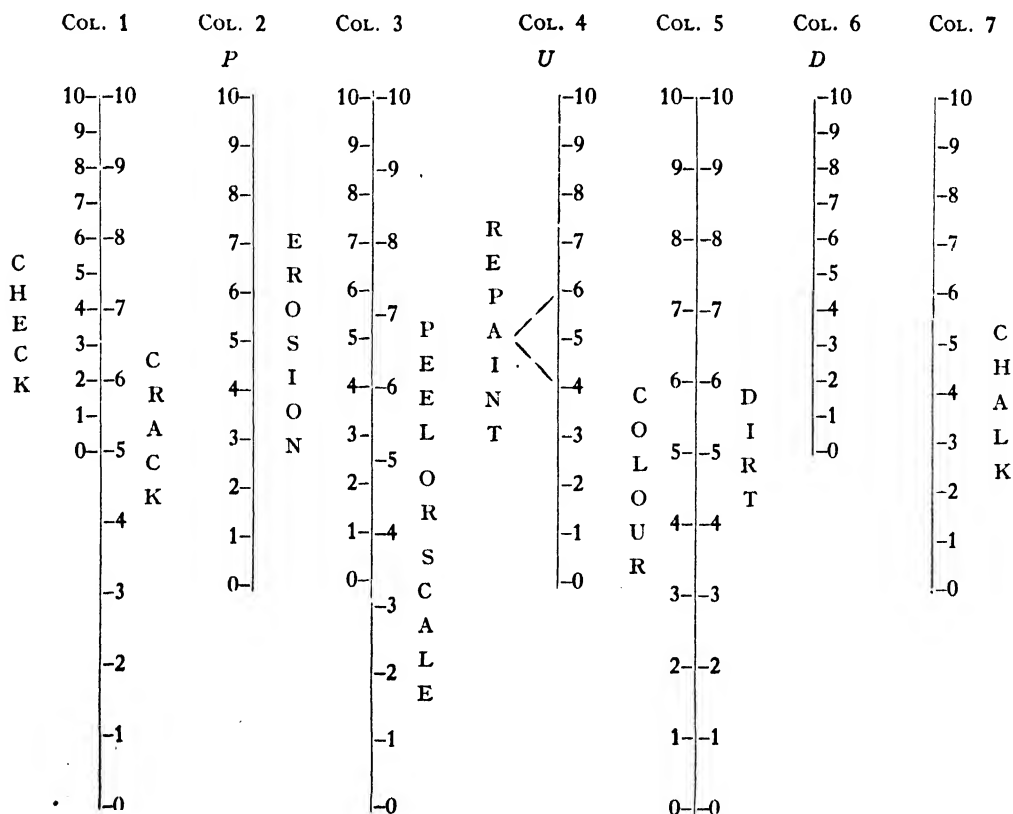


FIG. 1—NOMOGRAPH FOR ANALYSING TEST FENCE EXPOSURE DATA.



complete failure. Photographic illustrations of the degrees of failures of each type have been published in the form of a manual with the help of which the film under examination can be compared for various types of failures such as checking, cracking, peeling, etc., and for the degree of failures. Quantitative values may then be assigned for each type of failure. These figures although approximate are specific in nature and can be reproduced. It would be desirable if all the workers in this field in India, whether in industry or in research institutions, adopt this procedure and take photographs under typical Indian conditions. This will eliminate the *ad hoc* methods of recording paint failures.

Such pictures, however, do not indicate the ultimate worth of a paint film. A single variable, correlating all the types of failures will provide a useful measure. Garlock and McMullen<sup>4</sup> have evolved a nomograph for analysing test fence exposure data from which the durability of a paint film may be assessed by one single variable from the different numerical values for different types of paint failures<sup>6</sup>. The nomograph is reproduced in Fig. 1. It is established on the following basic relative values: crack is twice as serious as check; peel, scale or flake is twice as serious as crack; erosion is two-third as serious as peel or scale. Colour and dirt are equally serious, chalk is less serious than colour with high ratings and more serious with low ratings.

The method of using the nomograph is as follows:

1. If crack and check are both present, use the lower rating (column 1).

2. Find rating of erosion, scale or peel on appropriate scale (column 3).

3. Combine ratings on column 1 and 3 to derive P (column 2).

4. Combine the lower of dirt or colour rating (column 5) with chalk rating (column 7) to derive D (column 6).

5. Combine ratings P (column 2) and D (column 6) to derive final rating U (column 4).

The procedure will be clear by the following example:

Suppose by comparing the test paint film with the standards (photographs), the following values are obtained for degrees of failures; crack, 5; peel, 5; colour, 8; chalk, 7. The final rating (U) from the nomograph is 4½. As this figure lies between 4 and 6, the paint film is in a bad condition, and repainting is indicated.

## REFERENCES

1. HOLLEY, C. D.: *Symposium on Paint & Paint Materials*, A.S.T.M. Regional Meeting, Philadelphia, Pa.; March 1935.
2. BROWN, F. L.: *Proc., A.S.T.M.*, 1930, **30**, Part II, 852.
3. Federation of Paint & Varnish Production Clubs, Philadelphia, Pa., *Exposure Test Manual* (1941).
4. GARLOCK & McMULLEN: *Eagle-Picher Lead Company*, Cincinnati, U.S.A. (1947).
5. Federation of Paint & Varnish Production Clubs, *Official Digest* (1946).

## Symposium on Microwaves

AT THE INSTANCE OF THE *Radio Research Committee*, the *Council of Scientific & Industrial Research* has decided to hold a symposium on *Microwaves* on 8th and 9th October 1949 at Delhi under the chairmanship of Prof. M. N. Saha. The following 13 study groups, in charge of a specialist in each field, have been formed to make a comprehensive study of different aspects of the subjects and a large number of scientists are expected to present papers at the symposium:

1. Microwave Spectroscopy — PROF. S. K. MITRA
2. Radio Emission from the Sun — DR. H. RAKSHIT
3. Radar Meteorology — DR. S. C. ROY
4. Super-refraction — MR. M. R. SARWATE
5. Microwave Propagation and Electron Circuitry — DR. N. B. BHATT
6. Radar in Defence — S/LDR. T. T. JOSEPH

7. Effect of Different Types of Obstacles on Microwave Propagation — DR. S. S. BANERJEE

8. Semi-conductors and Crystals — DR. K. R. DIXIT

9. Microwave Dielectrics — PROF. P. C. MAHANTI

10. Nuclear Studies with Microwaves — DIRECTOR, INSTITUTE OF NUCLEAR PHYSICS, CALCUTTA

11. Thermal and Fluctuation Noise — DR. D. S. KOTHARI

12. Application of Microwaves in Television and Broadcasting — MR. B. V. BALIGA

13. Frequency Modulation — S. P. GHOSH

It has also been decided to hold on this occasion an exhibition of radar and microwave equipment in use in the various Government Departments.

# NOTES & NEWS

## Barium Chemicals from Barytes

THE Chemical Products Corporation, Cartersville, U.S.A., operates a specialized plant for the manufacture of barium chemicals from barytes. Low-cost production has been facilitated by the availability of good-grade barytes. Most of the plant is of the open-air type with only the finishing stages and storage under cover (*C.T.J.*, 1949, 124, 541).

The barytes is reduced to barium sulphide by the conventional coke-furnacing method and from this are produced 2 grades of barium carbonate and also barium chloride. The hydrogen sulphide liberated in the process is used for the manufacture of sodium and ammonium sulphides. The hydrochloric acid needed for the manufacture of barium chloride is made by chlorination of benzene, the products also manufactured being mono-chloro benzene and *ortho*- and *para*-dichlorobenzene.

Barytes used analyses to 96 per cent barium sulphate with 0.6 per cent iron and traces of other impurities. The ore is sized below 12 mesh, with as little of —200 mesh as possible. Extreme care is exercised in the maintenance of a reducing atmosphere in the kiln.

Two types of barium carbonate are manufactured: "free flowing" and "sulphur free". The latter material packs much more easily than the former.

In the manufacture of "free flowing" grade, flue gas from the boilers containing 11 per cent carbon dioxide is introduced into tanks containing barium sulphide liquor which has been filtered in a cast-iron plate-and-frame filter press. Steel equipment, with the exception of pumps and pipe-lines which are of Monel metal, is employed.

"Sulphur free" type is obtained by adding soda ash, in dry form, to hot barium sulphide solution. Addition is made until there is a slight excess of soda ash. The slurry is boiled and weak sodium sulphide solution is decanted off and filtered on an Oliver filter. The filter cake is repulped in water and finally washed on an Oliver filter free of sulphides.

A 20°Bé. barium sulphide solution containing the water-insoluble constituents from the kiln is used in the production of barium chloride. Hydrochloric acid is pumped in under slight pressure by a Duriron pump and acid addition is continued till a pH of about 5 is reached. More slurry is pumped in until the pH is 7 and all ferrous iron has been precipitated. The solution is brought to boil, decanted and filtered on an Oliver filter. All pumps, pipelining and fillings are either rubber-lined or constructed of Monel metal. Tanks are brick-lined, using an acid-proof cement.

The barium chloride solution is concentrated to 32°Bé. in batch-type evaporators (containing 30 per cent  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ), filtered through a wooden plate and frame press and crystallized in a batch-type vacuum crystallizer. The liquor is then pumped to a Monel vacuum pan and the crystals are dewatered and washed free of adhering mother liquor by cold water and finally fed to a rotary dryer. A low-temperature flame or hot air is employed to produce the crystalline type containing 2 molecules of water of crystallization.

## Fish-liver Oil Extraction Techniques

THE CANADIAN FISH-LIVER OIL industry adopts a number of processes for rendering oil from fish livers. Livers are rendered by the well-known process of cooking them by live steam and then skimming off the freed oil which rises to the top. At some plants, the "foots" remaining after the free oil has been skimmed off and, which still contain much oil, are hot pressed in canvas bags placed between wooden boards. The mixture of oil and water squeezed out (20 per cent oil) is then separated in settling tanks. Only low pressures can be used and the operation is slow. Another way of recovering oil from the "foots" is by alkali digestion (caustic soda or sodium carbonate) whereby the proteins are broken down and the oil is set free.

A novel patented process of cod liver manufacture practised on a

large scale is the "flotation" process. Livers, collected over a wide area in metal drums and filled with replaceable lids. The livers are preserved by the addition of "aquacide", which is a patented mixture of paraldehyde and sodium bicarbonate. This partly denatures the liver proteins and helps in the extraction process. This process is only applicable to fresh livers. The ratio of preservative to livers and the freshness of original livers are important factors.

After draining off the excess preservative, the livers are reduced to a very fine pulp, almost of colloidal dimensions, and passed on to a series of tall cylindrical flotation tanks, provided with stirring mechanism. The vessels are fitted with sight glasses and pipes for letting in warm water (120°F.) at various levels. It is claimed that the rate and time of addition of water and the speed of stirring are all critical factors. At first no free oil is seen, but suddenly the emulsion breaks up and free oil settles rapidly at the top, from where it is floated off. The yield is stated to be almost quantitative.

In another patented process raw livers are mixed with dried sugar beet residues using a paddle beater. The proportion is 13 lb. of beet residues, in the form of shreds, to 100 lb. of liver mince. Within a few minutes the mix is ready for pressing. The mixture is placed in cloth bags and pressed between wooden boards in a hand screw-press. The yield is equal to that obtained by steam rendering.

A novel process for removing water is by allowing the oil to fall in a thin film over a heated surface (170°F.) in a high vacuum. The time of heating is only a few minutes and the process is said to assist deodorizing also.

In one case alkali refining is carried out with solid magnesia, added to the hot oil along with a bleaching earth, combining the processes of neutralization and bleaching. Steam distillation under reduced pressure at a temperature of about 180°F. is employed to deodorize the oils. After bleaching, the oil is chilled to 32°F. and pressed to remove stearine.

Many firms render shark and dog-fish liver oils with live steam, but employ centrifuges instead of gravity settling to recover the oil.

In the case of halibut and lingcod-group of fish in addition to alkali digestion process, acid digestion and enzyme digestion are also

employed. Pepsin or papain is usually employed (*Food Manufacture*, 1948, 23, 359).

### High-strength Plaster of Paris

A. PROCESS FOR MAKING A LOW-WATER DEMAND (or low consistency), high-strength plaster of Paris of definite and controllable crystalline dimensions has been developed (*Ind. Eng. Chem.*, 1949, 41, 1061). The operation consists in autoclaving for 1 hr. or more, finely ground gypsum in the presence of 0.1 to 0.2 per cent soluble succinate, malate, citrate or maleate, removing the water and regrinding the product.

Although gypsum slurry, when heated in an autoclave, yields plaster of Paris, the product is extremely fluffy, has little strength when set and possesses an extremely high consistency. The addition of malates or succinates produces better results and gives products with improved properties.

Finely ground English gypsum (95 per cent through 325 mesh) of 99.9 per cent purity was autoclaved in a stainless steel gas-fired autoclave. The slurry consisted of 1 litre of water to 1 kg. of gypsum. Gypsum from various sources produced plaster of varying consistency and strength. The main impurity was limestone. The catalyst, sodium succinate, was found to be equally effective over the range 0.05 to 0.40; but at 1 per cent plaster made never took a hard set. The consistency of the plaster was minimum between 135°C. and 142°C. when English gypsum was autoclaved with 0.2 per cent succinate for 1 hr. Grinding has a considerable effect on the consistency and powder density of the plaster. 1 hr. of ball milling dropped the consistency from 59 c.c. to 41 c.c. and raised the powder density from 1.09 gm./litre to 1.29 gm./litre. Over 2 hr. of milling caused a rise in the consistency and lowering of powder density.

Plasters with compressional strengths as high as 3,300 lb./sq. in. have been obtained in the case of Oklahoma gypsum using 0.2 per cent succinate and heating for 1 hr. at 127°C.

### Fractionation of Starch

THE SEPARATION OF STARCH INTO its components, amylose and amylopectin, is now carried out by the use of thymol and cyclo-hexanol (*J. Chem. Soc.*, 1948, p. 1687). Thymol precipitates amylose from

its solutions, while amylopectin remains in solution. Cyclo-hexanol is recommended as a fractionating agent in place of thymol when the remaining component, namely amylopectin, is desired in a purer condition. Cyclo-hexanol-amylose contains a higher proportion of amylopectin than thymol-amylose.

In the thymol method, the starch is dispersed as completely as possible in boiling water to which 0.1 per cent sodium chloride is added and undispersed material is removed before the addition of thymol. The concentration of starch should not exceed 3 per cent in the presence of ions or 1 per cent in their absence. Powdered thymol, about 7 per cent on the weight of starch, is added to the starch solution and the precipitation is usually complete within 48 hr. The precipitate should not be left with the mother liquor for more than 60 hr. The amylose-thymol complex is in a more readily separable form if it is precipitated at 30°C. The amylose precipitate is best freed from its amylopectin solution by repeated washing with water saturated with thymol. Average specimens of thymol-amylose and amylopectin from potato starch show blue values of 1.0 to 1.2 and 0.21 to 0.23 and are hydrolysed by  $\beta$ -amylase to the extent of 96 to 99 per cent and 47 to 62 per cent respectively.

A 1 per cent paste of potato starch, to which a trace of sodium chloride had been added, was allowed to stand for 3 days at 30°C. in the presence of sufficient cyclo-hexanol to maintain a saturated solution throughout this period. The insoluble cyclo-hexanol-amylose complex was removed by passing the suspension thrice through a Sharples super centrifuge (25,000 r.p.m.). The amylose fractions were washed with water saturated with cyclo-hexanol. The blue values of average specimens of thymol-amylose and cyclo-hexanol-amylose are 1.1 and 0.9 respectively, the values for corresponding amylopectins being 0.22 and 0.18 respectively.

### Molecular Weight Determination Using Micro Quantities

A METHOD FOR THE DETERMINATION of molecular weights of chemical compounds using quantities less than 1 mg. has been described (*Can. J. Res.*, 1949, 27, 151). The method depends on the accurate measurement of

the difference in vapour pressure between a solution of the unknown compound and the pure solvent. This has been accomplished using a modified mercury U-tube monometer in a specially designed apparatus which can be easily fabricated in a laboratory. The results obtained indicate that the difference between the experimental and theoretical molecular weight is of the order of 2 per cent. The solvents employed should have relatively low vapour pressure and experiments should be carried out a few degrees below the room temperature.

About 0.5 c.c. of solvent and solution samples are employed. The sample-holders are placed in a thermostat and the samples are freed of dissolved gases by vacuum distilling ( $10^{-6}$  mm.) the solvent to the traps and back to sample-holders several times, using a suitable refrigerant to prevent loss of sample. Both solvent and solution were stirred by placing small, glass enclosed, *Alnico* magnets in the sample-holders and rotating a large horse-shoe magnet slowly under the thermostat. For ordinary work, precise thermostatic control is not required but it is necessary that both solvent and solution are kept at the same temperature.

The molecular weights of substances like mannitol, triphenyl phosphate and stearic acid have been determined using water, absolute alcohol and acetone respectively as solvents. An important consideration is the chemical stability of the solvent. Acetone in particular should be employed with care.

The method, as described, should be of considerable help especially where the amount of material available is limited. Once the apparatus is set up, the assembly is permanent, and a determination may be made in 2 to 3 hr. The samples are completely recoverable.

### Wire-screen Packing for Fractionating Columns

AN EFFICIENT, INEXPENSIVE WIRE-screen packing for fractionating columns is described (*Ind. Eng. Chem.*, 1949, 41, 1056). It is easy to fabricate, requires no welding, grinding, and precise fitting, or pre-assembly of parts. Ordinary metal or glass tubing with uniform bore can be used with the packing. The operating characteristics compare favourably with other types of screen-

packed columns which require much more skill, precision and expense in fabrication.

Columns of heavy-walled pyrex tubing, 0.625", 1" and 2" were employed. The wire screen was of stainless steel, phosphor bronze, brass or copper; the last 2 are less satisfactory because of their poor elasticity and tendency to relax under mild heat. 60×60 or 80×80 screen mesh was employed. Circular portions of the screen mesh are cut out, a portion of a circle being cut off from the edge of each disc of about one-third the diameter of the disc. This portion serves as a vapour passage in the assembly. The diameter of the screen discs should be 5 to 10 per cent greater than the diameter of the tube in which they are assembled. The close contact between the edges of the discs and the wall of the tube is important; otherwise liquid reflux would leak through and channel down the side of the tube.

The assembled unit or cell consists of 2 discs bowed in opposite directions (with their concave sides together) and having their vapour openings on the opposite sides of the column. In order to be doubly sure that a tight seal is obtained and that the cells are held firmly in place, split rings, similar to piston rings, are placed above and below each cell. These rings are made of stainless steel or phosphor bronze. Thus the assembled packing consists of a series of such cells so placed that each touches those above and below, providing a continuous screen surface over which the reflux flows and a zig-zag open pathway, through which the vapour passes.

Like other types of screen-packed columns, it is essential that the packing be thoroughly and uniformly wet before beginning a distillation. This is accomplished by a short period of boil-up under total reflux. Maximum plate efficiency was obtained at low boil-up rates.

### G.E.C. pH Meters

COMMERCIAL pH METERS FOR THE measurement of hydrogen ion concentrations with the aid of a glass electrode fall into two main groups, deflectional pH meters and potentiometric instruments. The deflectional pH meter has the merit of simplicity but has low accuracy, generally of the order of  $\pm 0.1$  pH unit. Furthermore, the calibration is liable to change with time and may be accidentally destroyed by over-load.

The potentiometric pH meters, however, may be calibrated to a very high degree of accuracy and will retain their calibration unimpaired by age or over-load. Unfortunately, potentiometric instruments usually have at least 6 controls requiring adjustment during measurement and increased accuracy could only be obtained at the expense of convenience.

The recent G.E.C. meters incorporate a patented circuit in which the number of controls needing adjustment during use has been reduced to 3, without sacrificing the high accuracy, permanence of calibration and robust construction of potentiometric instruments.

The new instruments are calibrated over the range 0.14 pH units with an accuracy of 0.01 pH unit for the portable model and 0.05 pH unit for the bench model.

Design of the instruments centres around a potentiometer circuit where the usual potentiometer resistance is controlled by 2 dials, one scaled in steps of 1 pH unit and the other subdivided to 0.01 pH. Indications are read through an aperture in the panel as a straight line of figures. An electrometer valve and two standard Weston cells are enclosed within the case and a sensitive galvanometer is mounted on the panel.

The electrodes of the hydrogen ion cell, usually a glass electrode and a calomel electrode, are connected to the two terminals.

Automatic compensation for changes in the E.M.F. of the glass electrode, due to temperature changes in the solution being tested, are compensated by means of the temperature compensator, which is immersed in the solution. This temperature-sensitive element consists of a boat of oxides, 0.05" in diameter and sealed into the drawn-out tip of a glass tube. This form of compensator has the advantages over other types that, owing to its small dimensions and heat capacity, it will respond instantaneously to temperature changes and can be used when only a few drops of solution are available.

In order to compensate for the changes which take place from time to time in the so-called "asymmetry potential" of the glass electrode, an asymmetry control is provided. If, when using a buffer of known pH value, an incorrect reading is obtained, the dials are set to the correct reading, and the asymmetry control is adjusted until the galvanometer is undeflected. Subsequent read-

ings will, in general, be correct for a period of 12 to 24 hr., after which a further change in the asymmetry potential of the glass electrode may occur.

The temperature compensator is normally connected in the circuit by means of a plug which is inserted in a socket on the panel. On withdrawing the plug, the circuit connections are automatically rearranged in such a manner as to convert the instrument into a millivoltmeter having a range of 1,400 millivolts and an accuracy of  $\pm 1$  millivolt. Within this range, no difficulty will be experienced in measuring the E.M.F. of a cell having an internal resistance of 1,000 megohms. (Contributed)

### Palm Candy Manufacture

A SIMPLE METHOD FOR THE PREPARATION of palm candy directly from palm juice is described (*J. Indian Chem. Soc., Ind. & News Ed.*, 1948, 11, 147). For its manufacture, the juice collected should not have undergone any fermentation. To prevent fermentation, 1 per cent formalin (10 c.c.) or 10 c.c. of 10 per cent lime emulsion is added for a pitcher holding 1 litre of juice. A mixture of 10 c.c. of 1 per cent formalin and 1 c.c. of lime emulsion has been found effective when the atmospheric temperature is above 30°C.

The juice is boiled in an iron or aluminium pan and concentrated to about half the volume. Any scum formed is removed and the concentrated juice filtered through a bed of activated charcoal (from paddy husk). It is boiled again to remove any scum that may be formed and filtered through a linen cloth or a cotton plug. Milk (1:5) is added to the boiling juice and temperature raised to 110°C. while stirring. The thickened mass is transferred to an enamelled can containing a few palm candy crystals. The depth of the juice in the can must be at least 3" to 4". The can is kept immersed in hot water and allowed to remain for 10 to 12 days. After this period the mother liquor is drained off and the palm candy crystals can be easily scraped off. The brownish crystalline and moderately hard candy contains 93 per cent sucrose and 1 per cent glucose.

### New Use for Gypsum

ONE OF THE RECENT INDUSTRIAL applications of gypsum is in the building industry. *Messrs Bell-*

rock Construction Ltd., Chertsey, Surrey, have devised an ingenious plaster panel which is both the building and the load-bearing unit (*Bull. Imp. Inst.*, 1948, 46 387). The panel consists of a honeycomb of hexagonal cells of gypsum plaster and fibre which is then faced on either side with a moulded gypsum plaster layer. It is the honeycomb reinforcement that largely contributes to the load-bearing capacity of these panels.

The panels used are of convenient sizes, i.e. are  $3' \times 4' \times 4''$  and  $5' \times 3' \times 4''$ . A wall for a single-storey structure can be rapidly built in two lifts. When two panels are butted together, the half cells along the vertical edges of both panels form hexagonal cells similar to those in the interior of the panels but, as the honeycomb core is slightly recessed from the edge, an open connecting passage is formed throughout this vertical row of cells. The edges of the adjoining faces of the panels and the base are sealed with plaster, using the thumb or a trowel. This seal sets in a few minutes and then liquid plaster is poured down the vertical connecting passage, completely filling all cells in the row and forming a solid vertical-keyed joint as well as a solid base plate bedding the wall to the foundation. The joints of the second lift are sealed and filled with plaster poured down the vertical passages; in practice the horizontal joints fill when the vertical ones are poured. The half cells along the top of the wall are also filled level with plaster, which forms a beam to carry the ceiling and roof. The poured plaster thus sets and forms columns and beams uniting the panels into a monolithic structure by a frame-work of solid elements.

To form a ceiling or flat roof, inverted T-joists of a suitable load-bearing capacity to bridge the span to be carried are laid, and the panels placed in position resting on the flanges of the T-joists. Alternatively, a supporting grid for ceilings or roofs can be formed by using panels with rebated margins to all 4 upper edges. When such panels are butted together, only the lower faces of the panels meet and the rebated margins form troughs, bordered by exposed half-honeycomb cells. Temporary supports are placed under the ceiling, and concrete is poured into the troughs

to form a beam frame-work, reinforcement being introduced as required.

By using suitable moulds, any desired form of ornamentation, picture rails, etc., can be cast on one or both sides of the panels. Panels can be readily sawn so that doors or windows of any size can be readily introduced even when the building has been completed. To fix doors and windows, wooden block or metal lugs are used to provide additional ties which enter the exposed cells in the panel sides. Joining edges are then sealed with plaster to prevent leakage and then liquid plaster poured in. For multi-storied structures, concrete or steel columns or beams calculated to carry the required load can be boxed within the panel.

Load-bearing test on a wall  $8' \times 9'$  and  $4''$  thick, consisting of two lifts each of two panels and a half panel at each side, joined horizontally with plaster and vertically with 3 non-reinforced concrete columns  $5\frac{1}{2}'' \times 2\frac{1}{2}''$  and provided with a reinforced concrete beam extending along the top of the wall, showed failure at 61.7 tons, equivalent to a loading of 6.9 tons per ft. run of wall.

In another test, a bungalow consisting of  $3' \times 4' \times 4''$  panels without any concrete reinforcing under a superimposed roof load of 20 lb. per sq. ft., gave a horizontal deflection of the walls less than 10 per cent of what might be considered an acceptable deformation in a load-bearing wall of this character. Thus, without reinforcement other than plaster, the system is particularly well suited to single-storey buildings.

Among the advantages claimed for this system of construction are speed of erection, low cost, good thermal insulation, and excellent fire-proof properties; also the ease with which electrical wiring and all fixtures may be applied. Very pleasing interiors can be obtained by incorporating in the unit-panel moulds, picture rails, skirting boards, tiles, panelling, or any form of decoration.

To allow for wear and tear from climatic extremes, a system of cladding with aluminium sheet has been devised.

The firm has developed a semi-portable gypsum calcination and grinding plant to produce a ton of gypsum plaster per hour for a fuel consumption of 5 gal. of oil per ton of plaster.

## A New Asbestos Product

PHOSPHO-ASBESTOS, AN INORGANIC thermo-setting compound consisting of crysotile asbestos fibre and orthophosphoric acid, moulded to the desired shape under heat and pressure, has recently won for its inventors the 1948 Edward Longstreth Medal of the Franklin Institute. According to *Asbestos*, October 1948, it was developed as the result of research to find a suitable material for such electrical devices as air circuit breakers, and it is claimed that this stone-like product is superior to compounds formerly used in respect to mechanical strength, resistance to moisture, dielectric strength and smoothness of surface. It can also be used for more intricate mouldings.

If it justifies the claims made for it, it should make possible the potential conversion of oil circuit breakers to air circuit breakers with the resultant increase in safety (*Bull. Imp. Inst.*, 1948, 47, 387).

## Insulating Board from Straw

A NEW PROCESS FOR MAKING insulating building boards from wheat straw has been recently developed by the *Bureau of Agricultural & Industrial Chemistry*, U.S. Department of Agriculture. This development promises to help straw to compete with sugarcane bagasse and wood-pulp as a raw material for insulating boards (*J. Franklin Inst.*, 1949, 247, 427).

The procedure consists of blending ordinary straw pulp used in making straw-board boxes and packing materials with a new type of pulp which is relatively less expensive and contributes increased strength and other desirable qualities to the product.

The new pulp is made by cooking the straw in water without chemicals at atmospheric pressure and running it through a "disc refiner" which breaks down the straw fibres into bundles of fine strands. The special properties of the pulp are due largely to the fact that half of its fibres are relatively narrow and slightly less than  $1''$  long. By blending the two pulps in varying proportions, it is possible to produce boards with a wide range of densities, greater strength at the same density than most commercial boards made from wood fibre and exceptional "impact strength".

## Ductile Cast Iron

BECAUSE OF THE INHERENT LIMITATIONS of properties obtainable, particularly ductility and toughness, cast iron is not satisfactory for many uses and the more expensive malleable iron or cast steel must be used. When magnesium is added to molten cast iron, the graphite in the cast iron is converted to a spheroidal form. This change greatly improves the strength, ductility and elastic modulus of the iron. Through this development, a low cost foundry iron may become available, which is easily produced and has excellent casting and physical properties hitherto found in cast steels.

The production is simple and can be applied to all iron compositions melted in the cupola or any other type of furnace. One method of addition of magnesium is the addition of magnesium-nickel alloys. This is operative with all basic types of cast iron including hyper-eutectic, ferritic and pearlitic, acicular, and austenitic varieties. The ductile cast iron is reported to have all the advantages of cast iron from the process viewpoint: low melting point, good fluidity and castability, ready machinability, and low cost.

Tensile strengths of 85,000 to 105,000 lb./sq. in. combined with some ductility were reported for pearlitic grades of cupola-melted ductile iron containing 3.2 to 3.6 per cent carbon and 1.8 to 2.8 per cent silicon. In contrast to grey iron, strength is only moderately affected by section thickness; ductile iron with 90,000 lb./sq. in. in a 1" section will ordinarily develop about 75,000 lb./sq. in. in a 6" section. Under stress the ductile cast iron behaves elastically more like cast steel than like cast iron, showing a proportionality of strain to stress up to high loads, with a modulus of elasticity of 25,000,000 lb./sq. in.

Simple annealing of the material is reported to develop ferritic structures which show up to 15 to 20 per cent elongation and 65,000 to 75,000 lb./sq. in. tensile strength. Normalizing and tempering have developed a yield strength of 116,000 lb./sq. in., a tensile strength of 127,000 lb./sq. in. and 2 per cent elongation. Quench and draw treatments and additional alloying can be used for higher levels of strength.

The ductile iron has improved endurance properties, much greater resistance to growth and oxida-

tion at higher temperature and excellent casting properties. It is as free in machining as grey iron of the same hardness but superior to grey iron having equivalent transverse or tensile strengths.

Magnesium should be added to a slag-free surface of molten iron at a time when the ladle is not being filled. The magnesium efficiency is said to improve if the ladle is covered during the reaction. The magnesium-iron should be poured promptly after treating, as the effects tend to disappear on holding. The addition of at least 0.4 and up to 1 per cent of magnesium is recommended, depending on the iron composition and the residual magnesium should be 0.03 to 0.10 per cent. Total carbon should be about 3.0 to 4.0 per cent and silicon 2 to 3 per cent. Addition of lithium, zirconium or calcium render magnesium ineffective, as does graphite after treatment.

Potential applications for this new material are extensive, particularly in automotive, railroad, and agricultural machinery, both in the as-cast and heat-treated form. Machine tools, crank-shafts, pumps, compressors, valves, and heavy industrial equipment could utilize its high strength and rigidity. Its ductility and probable consequent thermal shock resistance suggest superior performance in railroad car wheels and ingot moulds. Superior resistance to growth and oxidation has obvious values in engine, furnace, and other parts that are exposed to high temperature (*Ind. Eng. Chem.*, May 1949, p. 7A).

## Wattle Tannins

A METHOD EFFECTING ALMOST complete separation of wattle extract into tans and non-tans has been developed at the *Leather Industries Research Institute*, Grahamstown, S. Africa (*Ind. Chemist*, 1949, 25, 285). A yield of nearly 100 per cent of bulk quantities of wattle tannin containing less than 2.8 per cent impurities has been obtained. Work is also on hand to fractionate this purified wattle tannin into fractions of different molecular weights. Further researches in this field are connected with the difference between green and black wattle tannin. Analysis of the two types has shown that green wattle tannin averages 10 per cent greater in molecular weight than black wattle tannin. Previous work at the Institute confirms that limited use of bisulphite

liquors removes the colour differences and enables the use of green wattle for making satisfactory leather.

## Corn Protein Fibre

THE ONLY PROTEIN TEXTILE FIBRE marketed in America today is the one made from zein—the corn protein. The fibre is being produced by *Virginia-Carolina Chemical Corp.* (*Chem. Eng.*, 1949, 56, 154).

The fibre "Vicara" is produced as a soft uniform fibre, light-golden in colour. It is clean, free of noids and a crimp can be imparted to the fibre to facilitate carding and spinning. Its physical properties are: sp. gr. 1.25; toughness index 0.25 gm. cm./denier/cm.; shiftiness index 2.4 gm./denier/elongation; tensile strength 16,000 psi.; dry tenacity 1 gm. per denier. Vicara has an elasticity comparable to animal fibres, is resilient and resists deformation. It has warmth equal to the finest wool. Laboratory tests indicate that its insulating value is similar to that of animal fibres in a fabric of like construction.

Vicara is as water-repellent as wool and the water repellency is permanent at room temperature and is not removed by dry cleaning or repeated laundering. It is odourless and less inflammable than cellulosic fibres and resistant to mildew and bacteria and repellent to moth and carpet beetle larvae. It resists the action of acids and can be dyed with acid, chrome or metachrome colours, at boil, in the conventional manner used for wool dyeing. It is not easily attacked by alkali.

The fibre can be spun or woven on any conventional system and can be processed and handled on regular textile carding, spinning and weaving equipment.

## Antibiotic from Banana & Sweet Potato

TWO ANTIBIOTICS HAVE BEEN isolated from the pulp and skin of green and ripe bananas. One is active against fungi, specifically the fungus causing wilt disease of tomato plants. The other, apparently formed during the ripening process, is active against bacteria, and is found in the pulp and skin of ripe bananas only.

The sweet potato plant also produces two antibiotics, one of which has activity against fungi. It not only stops the growth of *Mycobacterium phlei*, a non-pathogenic relative of the tuberculosis



germ, but kills it (*Science News-letter*, April 23, 1949, p. 260).

### Jute Industry in Germany

*THE BIOS Overall Report No. 17* (H.M.S.O. London, price 6d.) deals with the jute industry in Germany during the period 1939-1945.

With the cessation of jute imports, alternative materials such as paper, regenerated cellulose and naturally occurring cellulosic fibres of European origin were employed as substitutes for jute. Excellent paper yarn was produced from high-strength Kraft from Scandinavia and indigenous unbleached sulphite paper. A typical product of the latter variety of 42 gm./sq.m. basic weight possessed a breaking length of 8·400 m. with a stretch of 1·5 per cent in the machine direction. A number of oil-soluble emulsifying agents, "Emulphors", were developed by the I.G. for opening and softening of jute. A mixture of 75 per cent mineral oil and 25 per cent whale oil was extensively used for oiling jute.

A report from I.G. deals with the use of colourless, water-soluble textile sizing agents based on polyvinyl compounds. Highly viscous, water soluble, clear, polymerization products of acrylic acid and its salts can be obtained by using pure colourless acrylic acid as the starting material. The properties of these products such as solubility, viscosity, and film-forming can be varied within wide limits by mixed polymerization with water-soluble and non-water-soluble vinyl and acryl compounds. By means of polymerization of vinyl compounds with hydroxyl groups or acid amide groups certain polymerization products are obtained which can be hardened by treatment with aldehydes. Similar products are obtained by saponification polyacrylonitrile polymers and mixed polymers. These products are of interest as sizing agents.

Phenols and their chloro and nitro derivatives were the chief agents employed for the protection of starch from mildew attack. The products have no affinity for the fibre and are easily leached out. Mildew-proofing agents, marked under the name "Preventols", most of which are chlorophenols, were frequently added to starch pastes. It is claimed that these products are not affected by reducing or bleaching agents.

The alteration and adaptation of jute-spinning machinery in

order to spin paper yarns was carried out extensively by the German jute industry. One interesting departure in the weaving of paper yarns was the weaving of a netting fabric on looms based on the chenille-loom principle.

An interesting device noted in connection with the sizing of the viscose staple fibre warp was an electrical device on the slasher designed to compensate for the yarn contraction which normally occurs at this stage.

Reasonably clean fibre, of good length and appearance, was obtained from green flax. The straw was run through multiple fluted rollers, followed by passages through low shakers and low scutchers. Green hemp was processed on a turbine scutcher. Jute softeners were employed to knock off the hemp bark; the final cleaning by means of a hemp hackler gave a 15 per cent yield of the fibre.

Of the rot-proofing treatments used, the cuprammonium process is the most important. A typical treatment consisted of padding the cloth through the cuprammonium liquor (24 gm. of copper per litre) at a speed of 5 m. per min., removing the surplus by scraping or squeezing, and drying in a hot chamber followed by drying cylinders. Bitumen and one of the oppanols (poly-isobutylenes) from benzene solutions were employed; a life of 10 years was claimed for the treated product.

A considerable amount of work was carried out by the I.G. on the waterproofing of textiles. A product marketed under the trade name "Akanstan N 1139" in which formaldehyde-bisulphite was used to render the finish more resistant to water, contained diammonium phosphate, 79 per cent, sodium formaldehyde-bisulphite, 20 per cent; and Soromin N (a mixture of stearethanolamide, 17 per cent; sodium sulphate ethanolamide of the fatty acids from coconut oils, 38 per cent), 1 per cent.

Hydrophobic metallic soaps were used extensively for the production of water-repellent finishes. Aluminium acetate, formate and triformate were all employed in the first bath, and the second bath consisted of a soapwax emulsion. Water-repellent finishes employing wax emulsions were also produced. It was stated that the higher the melting point of the wax, the better was the water repellency but the difficulty of obtaining a satisfactory emulsion was correspondingly greater.

A considerable amount of research was carried out on the infra-red reflectance of coloured textiles in connection with the subject of camouflage, and very few dyes showed high absorption. Hydron Olive GX was, however, exceptionally good. The subject is of interest to the jute industry on account of the employment of jute scrim and hessians as camouflage materials and the development of infra-red photography in the detection of camouflage.

Although the Bundesmann water-repellency test was developed in Germany, a number of other tests were preferred. One of these consisted of a water spray directed on to the fabric which was supported at an angle of 45° to the vertical. The spray fell 75 cm. from a viscose spinneret, and the time of use was 30 min. The water taken up by the fabric (found by weighing) and the time to pearl (i.e. the time when drops spread out on the fabric) were noted. The specimen was shaken to remove loosely adhering moisture before weighing. A second method consisted of the conventional hydrostatic-head principle, in which the water level providing the pressure was raised by 10 cm. per minute and the pressure at which the third drop penetrated the fabric was taken as a measure of the resistance of the material. A hydrostatic "bag test" was also employed for the testing of tarpaulins, canvas, etc. A bag of the material was formed and filled with water to a height of 30 cm. The material was satisfactory if no water percolated in 24 hr.

### Pest Control

THE FIRST REPORT OF THE WORK of the Pest Infestation Laboratory, D.S.I.R., London, has recently been published (*Pest Infestation Research*, 1947, H.M.S. Office, London, price 6d.). During the war, the work of the Laboratory was confined to the inspection of stored food and the application of appropriate control methods to prevent infestation. The work of the Laboratory involved biological studies of insects and physical studies of bulk grain.

One important outcome of the work of the Laboratory was the "carbon dioxide method" for estimating the infestation of a given sample of grain. This consists in bottling a sample and incubating it for 24 hr., after which the carbon dioxide content

of the air between the grains is measured. The grain itself produces a negligible amount of carbon dioxide but the insects produce a measurable quantity. The concentration of carbon dioxide is proportional to the number of insects present inside the grain.

It has also been demonstrated that insects cause grain to heat up through the formation of hot spots.

The work on the silo fumigation of grain was one of the many problems undertaken by the Laboratory. The work included studies of the penetration of the gas into the foodstuffs, its sorption—physical or chemical,—and the nature and permanence of residues in addition to the toxicity of the gas to many insect species.

Work on warehouse sprays resulted in the development of a method for pretreating the surfaces to be sprayed which gives a greatly increased duration of toxicity. The Laboratory was amongst the first to report the striking toxicity of DDT to houseflies.

### The Indian Dairy Research Institute

THE INDIAN DAIRY INSTITUTE, Bangalore, celebrated its Silver Jubilee on 8th July 1949. The function marks the completion of 25 years of useful service to the country by the Institute.

The Institute has developed from the old Imperial Institute of Animal Husbandry and Dairying which was started in Bangalore 25 years ago as a centre for dairy education and research. The establishment of the Institute was an important landmark in the history of dairying in India and this was the first practical step undertaken towards the organization of cattle breeding and dairying industry on scientific lines.

The Military Dairy Farm at Bangalore had a large dairy and the headquarters of the Imperial Dairy Expert was transferred to this centre in 1923 and was utilized as the main educational and experimental centre under the name "Imperial Institute of Animal Husbandry & Dairying". With the establishment of the Institute, a two-year training course for the award of Indian Diploma in Dairying was also started. During the years 1923-32, the activities of the Institute were mainly confined to the training of students. In 1931 the Institute was separated from the Indian Agricultural Institute and

was recognized as an independent Dairy Department by the Government of India. A scheme for the reorganization of the Institute was put forward in 1944, at the instance of the Government, which envisaged the expansion of the research sections, opening of a Dairy Science College and training of a larger number of diploma students. Some of these measures have been implemented already.

The Institute has carried on its pioneering work of training personnel and conducting research in dairy science and technology for the last 25 years with credit and has built up a tradition of its own. It has attracted students from all over India, Burma, Ceylon and Malay Federated States. The total number of students who have undergone training in the institute up to date is 759. Of these, 307 are diploma-holders and 162 postgraduate workers. Many of these scholars have made valuable contributions to dairy science and technology.

The Institute, at present, consists of 4 research sections, viz. Dairy Husbandry, Dairy Chemistry, Dairy Technology and Dairy Bacteriology. The total number of research publications, of importance to fundamental and applied sciences, to-date numbers 172.

The staff of this Institute have also taken a prominent part in organizing the Indian Dairy Science Association and in bringing out a scientific journal, *The Indian Journal of Dairy Science*, which has received international recognition.

The work of the Institute in the practical field is considerable and valuable. The Institute pedigree stock of animals, built up as a result of careful selection, have won a high reputation all over India. These animals are in great demand for grading up and for improving indigenous cattle. A centre for artificial insemination is being maintained at the Institute for the past 3 years and is responsible for a great deal of pioneering work in rural areas. The Institute has carried out work on the composition of milk of different Indian animals, enzymes and vitamins in milk and milk products, composition and storage of butter and *ghee*, *dahi*, *khoa* and other indigenous products, bacteriological quality of market milk and on the problems relating to clean milk production, microflora in *dahi* and butter, methods

of *ghee* manufacture, utilization of surplus milk and skim milk as cheese and *khoa* and problems of long-distance transport of milk from village to urban consuming centres, etc. These and other investigations have made this Institute a leading research centre in dairy science in the country.

### Tin Research

AN ILLUSTRATED BOOKLET PUBLISHED by the *Tin Research Institute* England, reviews the activities of the *Institute* during the year 1947-48. An important task recently completed is the completion of a booklet on hot-tinning, which will make readily available the most up-to-date information on all the processes involved. The hot-tinning laboratory, which has been recently equipped with modern industrial-sized plant, plays an important part in this field. Two machines have been designed which will stimulate the rapid installation of modern continuous strip electro-tinning plants. A new 88-page booklet summarizing up-to-date information on the composition, properties and methods of using solder has been published. The problems associated with the production of relatively small chill-castings have been solved and attention is being devoted to the continuous casting of bronze rods and tubes, and to the improvement of sand-cast bronzes. A comprehensive book on this subject is under publication. Yet another booklet issued by the *Institute* is on bearing metals, and describes the more important points to be observed in their manufacture to ensure good performance.

Corrosion stations, situated in 6 widely different parts of Great Britain and at 3 points in U.S.A., are being used to study the corrosion-resisting properties of tin and tin-alloy coatings under conditions likely to be encountered in service. It has already been shown that a thin coating of tin on steel is an excellent basis for paint and tin or tin-lead solder, and is now being applied for this purpose to the edges of motor-car body panels. New methods of applying tin locally to steel are being investigated.

Alloy coatings, containing 80 per cent of tin and 20 per cent of zinc, are being increasingly used for protection of steel in the electrical field where their good corrosion resistance and excellent solderability are proving advantageous. Electro-deposited speculum



plating, containing about 42 per cent of tin and 58 per cent of copper, has been found to provide an attractive finish for articles. Its advantages are a bright silvery-white appearance, resistance to wear and freedom from tarnishing. A new electroplating bath, which affords a wider range of current densities than has hitherto been possible with still solutions, is under investigation.

### Indian Association for the Cultivation of Science

IN PRESENTING THE ANNUAL report of the Association for 1948-49, Prof. P. Ray, Hony. Director, referred to the progress made towards the construction of the new research laboratories at Jadavpore. An important event during the year was the laying of the foundation-stone of the research laboratory building at Jadavpore by Dr. B. C. Roy, Premier of West Bengal.

The research activities of the three existing research departments of the Association's research laboratories, viz. X-rays and Magnetism, Optics, and Physical Chemistry, functioned as in the previous year. The Department of X-rays and Magnetism concentrated their investigations on the study of extra reflections in Laue photographs; X-ray study of the glass; study of the magnetic properties of single crystals and of solutions containing para-magnetic ions; study of electrical and magnetic properties of semi-conductors; construction of magnetic and electrical apparatus; and production of low temperature. Researches in the Department of Optics included the study of Raman spectra and *cis-trans* isomerism; Raman spectra of substances in different states; Raman spectra of organic vapours; fluorescence of organic compounds; structure of resonance line of mercury filtered through mercury vapour; Brillouin components in crystals; and luminescence and absorption of different varieties of diamond. The Department of Physical Chemistry conducted investigations on the following lines: fundamental studies on polymerization kinetics; inhibition and retardation of vinyl polymerization chain transfer by solvents in vinyl polymerization; cellulose chemistry including benzyl cellulose and acetyl cellulose; trial experiments on the preparation of alkyl resins from Indian raw materials; solubilization phenomenon; solubility and co-solvency

of metallic soaps; kinetics of halogenation of fatty acids; and dielectric behaviour of substances of high molecular weight.

Professor S. Chapman delivered the Ripon Professorship Lectures of the Association on "The Magnetism of the Earth" on January 14 and 15, 1949. The Mahendral Memorial Lecture was delivered on February 23 by Dr. B. S. Guha on "The Aboriginal Tribes and Their Places in Indian National Life".

The Association received from the Government of India a capital grant of Rs. 4,32,000/- towards the construction of the new research building, and a recurring grant of Rs. 2,66,700/-. The Council of Scientific & Industrial Research made a grant of Rs. 13,212/- for X-ray studies of metals and alloys and of Rs. 6,419/- for like investigations on coal. The Indian Physical Society contributed a sum of Rs. 700/- towards the Indian Journal of Physics.

The budget estimates of the Association for 1949-50 show a receipt of Rs. 2,95,000/- and an expenditure of Rs. 2,99,400/-.

The Woodburn Medal was presented to Dr. Shyamadas Chatterji for original research in Physics, *Nikunja Garabani Prize* to Sri Ranjit Kumar Sen and *Jatindra Chandra Prize* to Sri Ajit Dutta for best researches carried out in the Association's laboratories.

### Conservation Studies in American Universities

THE STUDY OF CONSERVATION of natural resources is receiving increased attention in the schools and universities throughout the United States. Many universities have added new courses, and specialized departments are being set up. One of the established leaders in the field is Cornell University (Ithaca, New York). Here a 4-year undergraduate course and specialized graduate training are supplemented by 2 general conservation courses open to the entire student body. The courses serve a dual purpose: (1) to establish a basis for advanced study for those who plan to make conservation work their career; and (2) to give educated laymen a foundation in the principles of conservation necessary to equip them to judge public issues wisely. Washington and Jefferson College, Washington, Pennsylvania, plans to establish a department of conservation next fall.

Soil conservation problems are of particular concern to agricul-

tural states. The University of Michigan maintains a professional school of forestry and conservation. Principles of soil, water and wild-life conservation are stressed at the University of Illinois. All students of agriculture are required to study conservation of natural resources. Marquette University, Wisconsin, Stanford University and Rhode Island College are a few others to have conservation courses on their curricula (USIS).

### Historical Statistics of the United States

A STATISTICAL SUMMARY OF American social and economic development since 1789, with figures for each year, is being issued by the U.S. Bureau of the Census, entitled *Historical Statistics of the United States, 1789-1945*. The book is the historical supplement to the annual *Statistical Abstract of the United States* which, for 70 years, has been the official statistical yearbook of the United States. This volume was prepared with the co-operation of the *Social Science Research Council*.

The volume provides business men, librarians, economists, teachers, and students with a statistical reference volume which brings together nearly 3,000 statistical time series of annual data, carried back to 1789, where possible. Fourteen broad subjects are covered. The book not only will provide data for immediate use but will serve as a starting-point and guide to original sources of data for those wishing greater detail, discussion, or explanation. The text provides definitions of terms and brief annotation, together with specific statements of sources. A special appendix contributed by the National Bureau of Economic Research provides monthly and quarterly figures, similarly carried back in time, for 30 statistical series which are recognized as useful indicators of business conditions.

The subjects covered are: wealth and income; population characteristics, immigration, and naturalization; vital statistics, health, and nutrition; labour force, wages, hours, and working conditions; agriculture, land, forestry, and fisheries; minerals and power; construction and housing; manufactures; transportation (including railroad, shipping, roads, and air transport); prices; balance of payments and foreign trade; banking and finance; federal finances, copyrights and patents, and state and local government

finances (*Foreign Commerce Weekly*, 1949, 35, 37).

### Development of Arecanut

THE GOVERNMENT OF INDIA HAVE decided to set up a Central Arecanut Committee, with the Minister for Agriculture, Government of India, as President, for the improvement and development of the production and marketing of arecanut and its products. The Committee will assist and encourage agricultural, industrial, technological and economic research in arecanut; produce, test, and distribute improved varieties of seed; encourage the adoption of improved methods of cultivation, assist in the control of parasites and diseases which affect arecanut; encourage the adoption of improved measures for arecanut marketing; give financial and technical assistance to organizations engaged in growing, processing, grading and marketing arecanut, establish a market intelligence service and recommend maximum and minimum prices to be fixed for arecanut and the controlled purchase and distribution of arecanut.

The Government of India will give an annual grant to the Committee not exceeding Rs. 5 lakhs and will appoint a Secretary and Co-operative Arecanut Marketing Officer to assist the Committee.

### Committee on Soil Science

THE GOVERNMENT OF INDIA HAVE decided to appoint a Central Committee on Soil Science with Dr. J. N. Mukherjee as the Chairman and 7 other members to guide and co-ordinate the scientific study of soil in the country to put agricultural production on a planned basis.

The functions of the Committee will be to advise the Government of India on matters of soil research and soil surveys; to guide and co-ordinate the scientific study of soil so as to ensure that soil survey proceeds on right lines; to lay down the lines of soil survey to be followed for purely scientific and specific utilitarian purposes, and soil survey training; to preserve in a central place the soil profiles representative of various types of soil; to provide for the interchange of information and samples regarding soil types amongst the workers within the country as well as in foreign countries.

Soil profiles from various provinces and states will be preserved at the *Indian Agricultural Research Institute*, New Pusa, and the work on the preparation of soil maps of India, according to the lines laid down by the Committee, will be entrusted to the Institute.

### Central Tea Board

A CENTRAL TEA BOARD HAS BEEN established under the chairmanship of the Hon. Mr. S. K. Sinha, with its headquarters at Calcutta. The Board will be composed of representatives of tea growers, exporters, the trade, labour, chambers of commerce, the central government and the governments of tea-growing provinces and states.

The Board will take over the activities of the Indian Tea Market Expansion Board, and arrange for research, collection of statistics, fixing of grade standards, improving of marketing of tea and bulk buying on behalf of government. The Board's activities will be financed from a cess collected on all tea exported from India.

### Journal of the Aeronautical Society of India

THE FIRST NUMBER OF THIS journal (quarterly) contains a short account of the inaugural meeting of the Aeronautical Society of India held at Bangalore on 27th December 1948 and an illustrated 12-page report of a lecture entitled "Progress in Aeronautics" delivered by Sir Ben Lockspeiser under the auspices of the Society.

The journal is well printed and neatly got-up.

### Announcements

**1851 Empire Exhibition Scholarship** — The 1851 Empire Exhibition Science Research Scholarship for the year 1949 has been awarded to Mr. K. G. Ramnathan, Lecturer in the Physics Department of the Indian Institute of Science, Bangalore. Mr. Ramnathan is the fourth research scholar working under Sir C. V. Raman to get this distinction.

**Mr. A. N. Khosla**, Chairman, Central Water Power, Irrigation and Navigation Commission, India, has been elected Vice-Chairman of the World Power Conference for a period of 3 years by the Executive Committee of the World Power Conference at its meeting held in Brussels on 27th June 1949.

**Indian Association for the Cultivation of Science** — The following office-bearers of the Council of the Association have been elected for 1949-50: Prof. M. N. Saha (President); Mr. C. C. Biswas and Dr. J. C. Ghosh (Vice-Presidents); and Prof. P. Ray (Hony. Director).

# Reports from States & Provinces

## MYSORE

### Survey for Mineral Deposits

A STATE-WIDE SURVEY FOR IRON, manganese and chromite ore deposits is being undertaken by the government. Manganese is available in Shimoga and Tumkur districts and chromite ore is reported in Hassan and Mysore districts. The survey is expected to yield definite information as to the availability and extent of low grade ores of these minerals.

## EAST PUNJAB

### Improved Wheat & Rice Strains

A NEW STRAIN OF WHEAT, RESISTANT to black rust, and a new strain of rice facilitating early detection of the wild rice strains growing in the Kangra Valley from the cultivated strain, have been developed by the Provincial Agricultural Department. The new wheat strain, which has been named C 217, has proved successful at the Ambala Experimental Farm and it is expected to do well in the sub-mountainous districts of Gurdaspur, Hoshiarpur and parts of Kangra.

Wild rice grows extensively in the Kangra Valley and has been a menace to rice cultivation in the area as it is impossible to differentiate it from the cultivated varieties in the initial stages. The new variety is a cross between a purple-leaved variety from Central Provinces and 2 indigenous varieties of East Punjab and enables early identification of the wild strain admixture.

## MADRAS

### Madras Institute of Technology

THE INSTITUTE WAS DECLARED open by the Premier of Madras on July 19. A munificent endowment of Rs. 5 lakhs by one of its sponsors has helped the Institute to start functioning at an early date. The Institute, which is

modelled after the Massachusetts Institute of Technology, will impart instruction in engineering sciences such as electronics, instrument technology, automobile engineering and aeronautics. The courses in motion-picture technology is deferred to a later date.

The Institute is situated in extensive grounds covering an area of 57 acres of land previously used by the Royal Indian Navy. Two huge hangars and other buildings on the site are being used at present for housing the technological block and for providing hostel and housing accommodation for the students and the staff.

### Wattle Plantation

THE GOVERNMENT HAVE SANCTIONED a scheme estimated to cost Rs. 11,05,000 for planting wattle over an area of about 6,000 acres on the Nilgiri Hills. The acreage cultivated would be 600 acres per year on a 10-year rotation basis and not more than 250 acres will be under wattle in any particular locality. The entire area of 6,000 acres, after 10 years, is likely to yield about 30,000 tons of wattle bark and is expected to fetch a revenue of Rs. 24 lakhs per annum.

### Mining of Lignite Deposits

A SCHEME FOR MINING OF LIGNITE deposits in the South Arcot district has been drawn up by Dr. Ghosh of the Geological Survey of India. The scheme refers to an area of 200 sq. miles in this district where mining operations will be started in the near future.

A new area of lignite deposits has been discovered near Cannanore in Malabar district. The deposit so far investigated are shown to extend over an area of a sq. mile. It is learnt that mining operations in this area are not complicated by the occurrence of any artesian springs as in the South Arcot deposits.

Extensive deposits of good quality of mica have also been reported from the Wynad district.

## Cotton Cultivation

THE DISTRIBUTION OF BETTER seeds, fertilizers, providing irrigation facilities and introduction of a summer crop in suitable areas are the main features of the government plan to increase cotton cultivation in the province. A seed multiplication scheme was commenced in 1948 and 3,100 bags of improved seed were distributed to be sown in an area of 908 acres. The crops grown with the improved seed is reported to be healthy and vigorous and it is estimated to yield an average of 1,000 lb. yield of cotton per acre.

Growing of summer cotton has been taken up in the South Arcot district and an area of 2,354 acres have been sown with improved cotton seed.

### Upper Pennar Project

THE PROJECT, FOR WHICH THE foundation-stone was recently laid, is the first of its kind to be undertaken by the government in the Anantapur district. The project is estimated to cost Rs. 77 lakhs and will bring under cultivation 6,000 acres of land in addition to supplementing water supply to several tanks. The estimated return on the capital outlay is 11.5 per cent. The project is expected to be completed in about 3 years.

## NEW DELHI

### Plastics Industry Development

A THREE-POINT PLAN FOR THE development of the Indian plastics industry has been recommended by the Plastics Development Committee appointed by the Government of India under the chairmanship of Mr. J. N. Roy.

The Committee considered the three urgent needs of the industry, namely of moulds, plastics powder and technicians. To meet the need for moulds, the Committee suggested to the Government of India to start in the proposed machine-tool factory, a section for casting moulds with a capacity of about 250 moulds per year for the present.

As regards plastics powder, the lack of uniformity in standards was stressed, which forced the Indian manufacturers to depend upon foreign imports. The industry was advised to develop an inspectorate of its own to inspect and grade the different products. The industry was further asked to develop an apprenticeship system for training of technicians, and to meet the difficulties created by conflicting demands from different manufacturers for technicians.

**Sugarcane Research**

THE GOVERNMENT OF INDIA HAVE appointed a Technical Committee to inquire into the cost of production of sugarcane in different parts of the country under the chairmanship of Sir T. Vijayaraghavachariar.

The Committee is expected to review the progress of sugarcane research and development and suggest ways and means of achieving quick results both in increasing the yield of sugarcane per acre and also the sugar content of cane with a view to bringing about an ultimate reduction of the cost of production.

**ERRATA**

This Journal :

Article entitled " German Dyestuff Industry ", 1949, Vol. 8, No. 4 :

Page 134 : for " Red G2H " read " Red GZH ".

" " for " Scarlet 3GL " read " Red 3GL ".

135 : for " Orange GCD " read " Orange GGD ".

" last line, for " AS " read " AS-BO ".

136 : for " AS-CR " read " AS-GR ".

" for " 2-Amino-4-Sulphobenzoic acid " read " 2-Ethyl-amino-5-sulphobenzoic acid ".

" for " Red LTR " read " Red ITR ".

**FORTHCOMING INTERNATIONAL CONGRESSES — 1949**

<i>Date</i>	<i>Subject of Conference</i>	<i>Organizing Body</i>	<i>Location</i>
Oct. 1-9	International Congress of Scientists concerned with Colonial Development	Ingenieurs pour la France D'Outre-Mer et les pays extérieurs, 11 Rue Tronchet, Paris 8e.	Paris
Oct. 3-10	Autumn Meeting of Institute of Metals	Secretary, Institute of Metals, 4, Grosvenor Gardens, S.W.1.	Paris
Oct. 17-Nov. 1	African Regional Scientific Conference	C.S.I.R., P.O. Box 395, Pretoria	Johannesburg
Oct. 23-28	Annual Seminar for the Study and Practice of Dental Medicine	Marion G. Lewis, Executive Secretary, 1618 Ninth Avenue, San Francisco 22.	Palm Springs, California
Oct. 23-30	22nd International Congress of Industrial Chemistry	Societe de Chimie Industrielle, 28 Rue St. Dominique, Paris 7	Barcelona
Oct. ( Undecided )	F.A.O., Meeting on Control of Infestation in Stored Grain	Food and Agriculture Organization	Palmira Colombia
Oct. ( Undecided )	F.A.O. Meeting on Livestock Breeding in Tropics and Sub-tropics	Food and Agriculture Organization	Cairo
Oct. 24-17 or Nov. 7-10 ( Undecided )	International Conference on Statistical Mechanics and Elementary Particles	University of Edinburgh	Edinburgh
Nov. 1-5	Pacific Chemical Exposition	James O. Clenyon, Research Association of California Research Corporation	San Francisco
Nov. 3-10	5th International Animal Husbandry Congress	Prof. Loroy, 16 Rue Claude Bernard, Paris 5e.	Paris
Nov. 16 ( Provisional )	Symposium on Metallurgical Applications of the Electron Microscope	Secretary, Institute of Metals, 4 Grosvenor Gardens, S.W.1.	London
Nov. 27-Dec. 2	Annual Meeting of American Society of Mechanical Engineers	American Society of Mechanical Engineers	New York
Nov. ( Undecided )	F.A.O. Annual Conference, Fifth Session	Food and Agriculture Organization	Havana
Dec. 16-18	10th International Ornithological Congress	.....	Washington
Undecided	3rd International Conference on the Chemistry of Cements	Bureau of Standards	Washington
Undecided	Conference of Geologists. INQUA IV	University of Budapest	Budapest
Undecided	Congress of Psychotechnics	Dr. Franciska, Baumgarten-Tramer, Thunstrasse 35, Berne, Switzerland	Berne

# INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the Gazette of India, Part II, Section 1, for July 1949.

## Organic Chemicals

39260. I.C.I. LTD.: Improvements in or relating to the manufacture of aminoguanidine compounds: *Introducing zinc into a suspension of nitroguanidine in an aqueous solution of ammonium sulphate below 30°C. and removing the sludge.*
38792. GAUDIN: Manufacture of organic substances containing sulphur and new substances resulting therefrom: *Reacting sulphur with an aromatic substitution derivative of propylene at a temperature of 150° to 250°C.*
- 39236, 39237, 39238. CANADIAN INDUSTRIES LTD.: Polymerization of 4-vinylcyclohexene dioxide: *Polymerizing in the presence of boric acid; polymerizing 4-vinylcyclohexene dioxide in presence of a catalyst; comprising polymerized halogen-2-butadiene 1, 3 and halogenated polyethylene.*
- 39370, 39371, 39372. I.C.I. LTD.: Manufacture of an 1:4 dicyano-2-butene: *Reacting 1:4 dihalo-2-butene with hydrogen cyanide under non-alkaline conditions; reacting 3:4 dihalo-1-butene or a mixture thereof with the isomeric 1:4 dihalo-2-butene, with hydrogen cyanide under non-alkaline conditions; reacting hydrogen cyanide with dihalobutene in presence of water, copper salt catalyst and a metal carbonate.*
39204. PARKE, DAVIS & Co.: Process for the manufacture of quinioline compounds: *Reacting 4-haloquinoline with aminomethyl substituted aminophenol.*

## Miscellaneous Chemicals

40643. SCIENTIFIC OIL COMPOUNDING CO. INC.: Improvement relating to compositions having fungicidal and/or fungistatic properties: *Heating water insoluble soap of a metal with a metal salt of hydroxy quinoline.*
39512. I.C.I. LTD.: Production of pesticidal compounds or mixtures: *Treating longifolene with chlorine.*
40549. SHAWINIGAN CHEMICALS LTD.: Improvements in briquetting: *Briquetting non-metallic inorganic material by heating at an elevated temperature below the melting point.*

## Medical Research & Practice Including Clinical Application of Drugs & Pharmaceuticals

40507. LEVER BROTHERS & UNILEVER LTD.: Improvement in or relating to the purification treatment of fatty oils containing vitamin A: *Solution of oil in a non-polar hydrocarbon is passed through a column of activated gamma alumina and thereafter the oil is separated from the solvent.*

## Metals & Metal Products

38746. JOHN MILES & PARTNERS (LONDON) LTD.: Smelting of iron and steel: *Fluid containing*

*70 per cent oxygen and above blown in, in a low shaft furnace.*

40898. ROVAL LTD.: Anodic oxidation of aluminium and its alloys: *Including in the electrolytic bath of an addition agent consisting of 15.6 per cent mineral matter, 80 per cent organic matter, 3.5 per cent moisture, chief mineral matter being calcium lignin sulphonate and organic constituents being dextrose, xylose resin, acetic acid, tannin and gums.*
40939. N. V. PHILLIPS' GLOEILAMPENFABRIEKEN: Magnet cores for electromagnetic devices: *Magnet core comprising a manganese-containing ferrite sintered to compactness with a Curie point higher than 250°C. and a ratio Fe : Mn between 4.0 and 1.5.*

## Stone, Clay & Glass Products

41015. THE TATA IRON & STEEL CO. LTD.: Manufacture of refractory material: *Employing calcined fire-clay alone or in admixture with non-plastic fire-clay.*
40757. INTERNATIONAL GENERAL ELECTRIC CO. OF NEW YORK LTD.: Method and apparatus for coating hollow glassware: *Directing a stream along the surface.*
40773. SHRI DHAR: Manufacture of hollow glass beads: *Forming glass bead tube from white transparent glass and then coating the inside of the tube with liquid bright gold.*
38564. J. LEICHTENSCHLAG: Seed drilling attachment to single-furrow ploughs: *A seed drilling attachment for a plough comprises a seed reservoir with a trap door that is opened at regular intervals by pins fixed to a wheel attached to the plough and rotated by the progress of the plough.*

## Miscellaneous

40148. JOSEPH SANKEY & SONS LTD.: Apparatus for use in determining the characteristics of coated metal sheets: *Comprising relatively movable platens and means incorporated with the machine for measuring and indicating the thickness of a specimen.*
39990. KAPUR & JAIN: Waterproof emery cloth or paper: *Process of manufacture comprises applying adhesive material containing a fungicide in molten state to basic cloth already treated with mildew resistant fixing abrasive material and stoving, thereafter applying coat of adhesive solution and stoving further.*
38170. DIOTTI, & S.A.I.C.I. SOCIETA ANONIMA AGRICOLA INDUSTRIALE PER LA PRODUZIONE ITALIANA DI CELLULOSA: System for utilizing the components of the reed (*Arundo donax*) differing from the cellulose possibly designed for the production of industrial cellulose: *Preparation of sugar juice, alcohol and fertilizer.*

# Helping the Indian Inventor

R. B. PAI

*Council of Scientific & Industrial Research, Delhi*

THE average Indian inventor is in a sad plight. Only a few of our men with inventive talent can be absorbed by large research institutions. For the most part, the inventor is left to his own resources which, so far as facilities for the commercial development of inventions are concerned, are extremely meagre and inadequate.

Hitherto the inventor has looked either to the Government or to big capitalists for help and guidance.

Government is already financing a good deal of organized research and the problem of furthering the utilization of inventions and patents arising from State-financed research is itself a difficult problem with which the Government is likely to be pre-occupied for many years to come.

The average capitalist is so used to getting easy returns from well-known and commonplace types of investment activity that he is not likely to divert his funds to the highly speculative and hazardous business of promotion of new inventions.

The inventor has, therefore, to look to other sources and take effective action suited to the circumstances.

The object of this note is to discuss a novel plan which should meet exactly the situation hereinbefore discussed. The plan will not appear fantastic or unworkable when it is pointed out that the principle of the plan has already been tried out on a vast scale under the auspices of the Canadian Government and with unexpected success. Details of the Canadian plan and its achievements and possibilities are discussed in the course of an article on the "Organization of Scientific and Industrial Research in Canada" by C. J. Mackenzie, President, National Research Council of Canada.\*

The plan for India, which will now be described, is in no way a copy of the Canadian plan and the only similarity between the two is that the germ of the two ideas may be treated as identical.

The plan in brief will be :

*The Preliminary Step* — To organize the inventors into a Central Inventors' Institute.

This is the first essential preliminary step to the main plan. The Government's interest in it will be only to afford recognition to the Institute as a warranty of the latter being organized along right lines. In fact, the Government may set the ball rolling by inviting the inventors to get together. Once this is done, the rest should be left to the inventors themselves, subject to the Government acting as a referee. In the first instance, to avoid bogus persons coming up, only those may be permitted to call themselves inventors for the purpose who have at least one subsisting patent to their credit. The Institute should be organized on entirely democratic lines and the members should choose an Enterprise Council.

*The Main Plan* — The most vital source of progress in a democratic state is its body of public-spirited citizens. It is this source that must be drawn upon if our industry is to develop on solid foundations. No doubt, the State and the big industrialists have most important parts to play. But an even broader approach is called for to base industry on truly democratic foundations. The underlying idea of the present plan is to enlist the sympathies of the ordinary public-spirited individual citizens by giving a practical shape to such broad-based support and encouragement. The plan would work as follows.

Initially some funds would be necessary to develop and commercialize the inventions of our private inventors. This fund is to be built up not by Government grants or big endowments by well-to-do philanthropists, but small amounts donated by ordinary citizens for a cause in which they have faith and which they believe would be conducive to the industrial progress of the country. These donations may consist of small contributions, no amount being considered too small; but the collections would come from a very large number of citizens. The idea would thus bring the very masses

\* *J. Sci. & Ind. Res.*, 1946, 5, 50.

into direct contact with the scheme and the interest taken by them would help to build up enlightened public opinion on the question of helping the Indian inventor. The contributors need not look up to any return other than the satisfaction of having made a small contribution for the building up of Indian industry based on *Indian inventions*; and even in this they should be prepared for some initial disappointments.

The Enterprise Council should issue a nationwide appeal, which should be endorsed by the Government, inviting donations to create a fund which will be utilized to develop as many Indian inventions as possible on a commercial scale.

This idea of tapping the resources of the general body of public-spirited citizens to create a fund for venture capital is the crux of the whole plan.

The Enterprise Council should work out a priority list from the inventions placed at its disposal and utilize the donated fund for the purpose of developing and commercializing the selected inventions. In the case of some of these inventions, further experimentation or trials on a pilot-plant or semi-commercial scale may be necessary. The Enterprise Council should finance and organize such trials. In certain other cases it may be possible to initiate commercial production without further ado.

The Enterprise Council should go ahead with daring combined with judgement. A premium should be placed on venturesomeness, and orthodox considerations of "soundness" should not be allowed to result in stagnation or sloth. Ideas must get moving and goods must be delivered. All that should be expected of those constituting the Enterprise Council should be that they do their job to the best of their ability and judgement. They should be answerable to the nation but nothing more should be expected of them than having done their very best.

There can be no doubt that if the organization is built up on sound lines as suggested above, the public will come forward with money and patronage. The plan will appeal to their highest national sense. If, for some reason, the experiment results in the proverbial mouse, nobody loses more than the small or big amount he voluntarily donated, knowing full well the chances involved. But failure is inconceivable. It will have a bad psychological effect. On the other hand, the chances of success are most alluring and the game will surely be worth the trial. There is every hope that the returns from the successful ventures will amply replenish the initial funds, reward the inventors and add to the nation's wealth and prestige.

## Engineering Exhibition in Delhi

AN ENGINEERING EXHIBITION IS TO BE HELD in New Delhi to coincide with meetings of the International Commission on Large Dams and the World Power Conference in India in 1951. Exhibits will include working models of major river valley projects, fixed models

of engineering devices, hydraulic models, models relating to power generation and utilization, land reclamation, soil conservation and cement factories. Overseas countries have expressed their willingness to participate.

## Metals in Relation to Living Standards in Industrially Under-developed Countries\*

**M**ETALS form one of the earth's most important resources in the support and enrichment of human life. In ancient and medieval times, metals helped man directly in his various avocations, his arts and crafts, raised his standard of living and lent grace to life by their use in the fine arts. Modern man today needs metals as much for the munitions of his destructive wars as for implementing human welfare in daily life.

The depletion of the earth's store of metals has increased progressively since 18th century but, with the 20th century era of world wars, it is going to become one of the biggest problems of the next century. Since 1914 more basic metals have been used up (a large part either destroyed or irreclaimably locked up) than during the whole of human history. The diminishing reserves of metals such as tin, lead, zinc have already become a problem. The 18th International Geological Congress, held at London in August 1948, devoted a special section to the discussion of "The geology, paragenesis and reserves of the ores of lead and zinc" which brought out this feature in relief. Doubtless, in some measure, the substitution of plastics and alloys of the light metals which are in more abundant supply and the discovery of new reserves in the remaining unexplored countries, together with resort to deeper mining and developments in the practice of ore-refining and upgrading, may put off the scarcity for two or three generations, but the lesson of metal depletion from the accessible parts of the earth's crust should be taken as nature's grim warning that man is spending away a prime treasure which is non-replenishable.

The critical shortage in metals is most felt up to now only in respect of tin, lead and zinc, but signs are apparent that accessible deposits of copper, nickel, manganese, wolfram and antimony are diminishing and new discovery is not keeping pace with demand. The extractable stock of platinum, silver and gold, left for future needs within minable depth, is becoming meagre. The situation, though general for the whole world, however, is becoming acute in the Western Hemisphere, chiefly the countries around the North Atlantic border where modern standards of life demand an inordinate use of metals; for the Eastern countries whose consumption of metals is comparatively insignificant and who have only played the rôle of miners of tin, manganese, wolfram, antimony and their accessories for wholesale export to the West, the situation is not so grave. This disequilibrium in the world's mineral economics calls for adjustment by wise and equitable solution and not by resort to the old policy of colonial exploitation, tariff barriers, etc., against weaker nations.

### Trends in the Use of Metals

Since the advent of the industrial era, the under-developed countries of the world have been exploited for their metals and ores by the industrially developed countries. Tin, manganese, wolfram and antimony have thus flowed in increasing volume from Malaya, Siam, India, Burma, China, Indonesia to Europe and North America. The *per capita* consumption of these metals in the producing countries has been negligible, and to the extent to which their mineral production has helped the development of metal industries of the world since

\* Paper by D. N. Wadia read at the United Nations Conference on Conservation and Utilization of Resources at Lake Success, August 1949.



1850 must be measured by their share in the spread of the industrial civilization of the last century. But the material return obtained by these countries for this service has been disproportionately small in most instances. This factor will tend to restrict, in the coming era of self-determination for each nation, the flow of minerals and metals in future, except for more substantial gains. A growing trend towards conservation of mineral resources in hitherto backward countries and their utilization (or barter) for national benefit will be the most significant development of the decade. The natural result of this may be that the large engineering and manufacturing industries of the world will not be able to draw, as unrestrictedly as in the past, on ores and raw materials except on the basis of reciprocity and on more liberal terms of exchange with manufactured goods which these backward countries need in building up more healthy national economy and standard of living.

India has supplied 40 million tons of manganese ores of the higher grade since 1900 at a price but little above the cost of mining and transport to the consumer; Malaya, Indonesia, Siam and China have supplied  $5\frac{1}{2}$  million tons of primary pig-iron during the same interval, and continues still to furnish the major part of the world's demand for this essential metal, for which no substitute is in sight. The largest supply of the strategic metal, tungsten (78 per cent), has come from China, Burma and Malaya ever since the discovery of its high potential in war and defence. China's mines supply 71 per cent of antimony metal, maintenance of which is vital to a number of industries. In a similar category must be placed the regular pre-war exports of over 2 million tons of iron ore to Japan, an industrially developed country, by Malaya and other undeveloped countries. Ceylon has supplied  $1\frac{1}{4}$  million tons of superior graphite during the last 60 years.

#### **Lack of Policy of Conservation**

None of these countries had any mineral conservation policy in the past and mining practice was unscientific, uneconomic and inefficient. A national conservation policy must imply complete and accurate appraisal of resources by organized resource surveys. With the exception of India, where an official geological survey has been in existence for nearly a century and which has since the war been quadrupled, and to a less extent in

China, Burma and Malaya, no scientific agency for mineral resource estimation, sampling and geological exploration on modern lines has been in working in Asian countries till very lately. The technique of aerial survey and mapping, geophysical investigation at depth, statistical control in resource assessment and mining, are being gradually applied in India after decades of experience of unsound mining and despoiling of depletable and non-renewable resources for export purposes.

But new trends are appearing in the post-war years. India has now a Bureau of Mines which is planning a programme of detailed appraisal of mineral deposits with systematic testing, stoppage of haphazard mining, ore beneficiation and use of statistics in enforcing conservation by local processing and utilization. Education and training of technical and mining personnel for putting into practice the chief elements of this programme is undertaken since 1945. It is only a matter of time for other countries of south-east Asia, which have metalliferous minerals, to follow in the wake of this programme. In these countries the pressure of increasing population, rising living standards, especially of food and public health, is creating an insistent desire for proper development, conservation and utilization of their mineral wealth, viz. use of new techniques and effective controls on export of raw products in place of the *laissez faire* attitude of the past. Everywhere there is a sustained drive to bring into use mineral resources as yet untapped to build up, with the revenues obtained from this source, more productive and diversified economies. The idea has slowly dawned that national resources must be cultivated for enriching the life of the people by judicious use and conservation.

The general lack of trained geological and mining personnel, technicians, engineers and other experts in the specific fields is being made good slowly by an intensive educational effort in training in local institutions, overseas scholarships and engagement of foreign experts on contract.

#### **Extent of Metal Resources of India & Countries of the Far East**

The resources of India in certain metals and ores are fully adequate to support higher living standards for the masses which is the aim of national planning. They are of a scale to ensure supply of indigenous needs for centuries and leave an exportable surplus

margin. The estimated deposits of iron, manganese, aluminium, titanium-ores and of the ferro-alloy metals, chromium and vanadium, give promise of India becoming, in the foreseeable future, an important metallurgical centre for ferrous metals and alloys and for the light-metal alloys. The strategic metals, thorium and beryllium, have been located in deposits of considerable size. Against this, the position in respect of non-ferrous metals is one of scarcity. The position with regard to lead, zinc, copper, cobalt and nickel may, in part, be improved by a more detailed exploration in the future. But, in respect of tin, silver, platinum and mercury, however, the deficiency is of a more permanent nature. A few notes on the existing proved and prospective metal resources of India with statistics of production and reserves are given in a following paragraph.

Burma has notable resources of wolfram and ores of lead, zinc, tin and nickel; China is believed to have the biggest iron reserves in the Western Pacific region, next only to the vast reserves of Singhbhum-Orissa in India, while her known tungsten and antimony reserves are yet of considerable magnitude. 60 per cent of tungsten and 71 per cent of antimony of the world is mined from these two countries from deposits which are regarded as ample for future needs. Malaya has moderate supplies of magnetic iron-ores, the chief source of supply to Japan. In tin, the south-east Asian countries — Malaya, Indonesia, Siam, China and Burma — virtually dominate the world production, though both production and distribution of tin is long controlled by the International Tin Cartel, which has prevented some of these countries from reaping the full benefit of their valuable natural asset. An indispensable accessory to metallurgical industries is graphite, for which Ceylon holds big reserves of pure crystalline and flake graphite, enough to last the world for a long period.

#### Outlook for Development & Potentialities

Expansion of these resources by discovery of new, or partly explored, or suspected deposits in the industrially under-developed countries named above is distinctly promising. On the other hand, the promise is limited by the difficulty and cost of the new methods of exploration, geophysical and geochemical, and insufficiently trained personnel. These features will retard the rate of discovery of new resources. These adverse

factors, however, have, in the past, proved to be not wholly disadvantageous, as they were the very factors that tended to conserve the underground resources of many undeveloped countries from too rapid depletion. In the race for export of ores, for which no indigenous market existed and the inducement of small immediate gain, the majority of the metal resources of these countries were mined haphazardly and unsystematically, the last consideration being given to any aspect of conservation. In countries which mine principally for export, this is the only aspect of conservation that needs effective safeguarding today, as it is the most serious threat to the world's mineral economy. The other aspects of metal resource conservation, viz. conservation of ore milling and concentration and conservation by substitution, reclamation, utilization of scrap, etc., are problems of the future when the mining industry has reached the second stage, viz. manufacture of the products of mines.

#### Metal Requirements for Industrial & Agricultural Developments

Accurate data and statistics of metal and ore production for export as well as for local consumption are unavailable for most Asian countries. Whatever ore is mined (and a considerable aggregate quantity as well as variety is mined) is mostly for trade purposes, for export overseas. The return for this is principally in the shape of consumer goods of general description and very subordinately in the form of processed or fabricated metals. The *per capita* use of metals, thus, in the every-day life of the people of these countries is not ascertainable through official statistics, beyond the knowledge that it is yet abnormally small. A trend for increased use of metals is slowly setting in, not for domestic or private life of the citizens, but for such general utility purposes as public works, engineering, railways, river navigation, etc.

The use of metals in agriculture in all the Asian countries is limited to a few primitive iron tools which the village smith fashions out of the local ores with forest fuel. All through the ages, man in Asia has made little use of metals in industry and agriculture, but he developed a remarkable skill in fashioning and alloying the metals, iron, copper, zinc, silver and tin, for his domestic utensils, the most obvious military weapons and articles of fine art. Ancient India acquired high reputation in the metallurgy

of iron and steel, copper, zinc and bronze. The fame of the ancient Indian steel *wootz*, in demand in Europe for sword blades, the 4th century iron pillar of Delhi, a solid shaft of rustless wrought iron, 16" diameter and 23' 8" high, and the ornamental bronzes and statuary of the 5th to 10th centuries are examples.

It would be, perhaps, too much to say that the comparatively small productivity of Indian agriculture in cereals, livestock and commercial crops is related to the small use of metals, or that more use of metals would mean better tillage. The age-old peasant traditions in India lend no support for these presumptions; at the same time, on a broad view of the problem, metals and machines are a *sine qua non* of large-scale agricultural practice, collective farming, reclamation of waste land, swamps and forests. Mechanized transport would bring fields, farms and factories nearer to markets. These are obvious facts, but rural and agricultural India, divided into 700,000 villages, reacts slowly to new forces.

Signs of mechanization of agriculture in some Asian countries are already becoming visible and the tractor-plough might become as popular in time as the motor-bus in the less remote parts, at any rate, of rural India. In industry, the pace is much quicker and the demand in India for pig-iron and steel, which used to be satisfied with about 2 million tons of the former and 1 million tons of the latter, put out by the Indian iron and steel plants in pre-war days, has increased fourfold at the present day. Better housing, road-building, motor transport, electrification programmes, cottage industries and the spread of air-conditioning in tropical climates will continue to swell these demands for metals in time. These, with heavy engineering plant and machinery required for the various multi-purpose water-power, irrigation and industrialization schemes which India and several Asian countries have launched since the war, will bring about a revolutionary rise in the East's demand for fabricated metals from a few hundred thousand tons to several million tons. To a limited extent, the indigenous resources of the Asian countries will be able to cope with these steeply rising needs in metals but a good part will have to be satisfied with imports from abroad. This demand will have to be adjusted against their capacity and willingness to continue to supply raw materials to the more advanced industrial

countries. To foster such interdependence of countries on the world's material resources and thus attempt to establish an equilibrium between these two sets of countries into which the world is unhappily divided today should be the goal of the United Nations Economic and Social Council. A desire for a friendly and satisfactory adjustment of this demand and supply for metals and minerals in meeting each other's wants and deficits in the coming years will be the best guarantee for the war-damaged world's peace.

#### Principal Metal Reserves in India, Production Statistics, etc.

Data and statistics regarding the following twenty metals found in deposits of workable size in India are given below:

**Aluminium** — Ore reserves are computed at about 250 million tons ( $\text{Al}_2\text{O}_3$ , 50 per cent); the deposits are fairly widespread throughout the country, in Bihar, Central Provinces, the Western Ghats, Kashmir and Madras. The annual production of aluminium in India is yet small; 4,000 tons, against a requirement of 15,000 tons.

**Antimony** — A deposit in Pakistan (Chitral) supplied about 1,000 tons of ore per year during the war. The smelting and refining was done at Bombay. China is the world's largest producer of antimony (about 16,000 tons in pre-war years), the largest deposit being found in the province of Hunan. The ore is smelted near the mines.

**Beryllium** — During the war, India exported considerable quantities of beryl. Beryl from Rajputana and Bihar is prized because it contains 11 to 14 per cent of  $\text{BeO}$ . The exports of beryl have been prohibited since 1946. Research on the isolation of beryllium and beryllium oxide is being carried out by a committee of the Board of Scientific & Industrial Research.

**Chromium** — Chromite is produced in Bihar, Orissa and Pakistan (Baluchistan); the last locality possesses large reserves. The production has been around 50,000 tons per year, the bulk of which was exported. Possibilities of upgrading lean chromite ores and manufacture of ferrochrome are being investigated.

**Cobalt** — The only area within the geographical limits of India which has reported deposits of cobalt is Nepal but the geology and probable reserves are not yet investigated.

**Copper** — The chief producing area is Singbhum (Bihar) which has an annual

output of about 6,500 tons of metallic copper. Other reported deposits are under investigation. A number of abandoned sites where a copper industry flourished till a century or two ago are known; these supplied all the needs of India in copper and brass in medieval times. Burma produced copper matte (about 13,000 tons in the pre-war years) but the ore reserves are not large. Annual consumption of copper in India is 40,000 tons.

*Gold* — Annual production averages 300,000 fine oz. The gold mines of Kolar (Mysore) are now 9,600' deep, the deepest metal mine in the world. The ore persists at this depth, but the problem of rock-bursts is increasing in severity.

*Iron* — India's resources in iron-ore are of vast extent. In the district of Singhbhum and adjoining parts of Orissa, the reserves occurring at surface are computed at over 8,000 million tons (metal content 60 to 65 per cent). The reserves in the Central Provinces and parts of Madras are also large. In contrast with the vastness of ore reserves, however, the annual production of pig-iron and steel yet remains small (2 million tons of pig-iron and about 900,000 tons of steel). New steel plants are being installed and the manufacture of a range of ferro-alloys is being considered.

China's iron-ore reserves are only next to India's, though they have not been fully investigated and the production yet remains small.

*Lead* — Production of lead in India at the present day is insignificant, though two centuries ago considerable amount of ingot-lead was produced in several parts of the country, mainly for military requirements. India's annual lead import is about 8,000 tons.

Burma produced 75,000 tons of ingot lead in pre-war years. Reserves of lead-zinc-silver ores at the Bawdwin Mines are calculated at 4 million tons, containing 16 oz. of silver to the ton.

*Magnesium* — Magnesite deposits of large extent and high degree of chemical purity occur in Madras. In Salem the estimated reserve is 90 million tons, analysing over 96 per cent of magnesium-carbonate. A considerable part of the annual output is exported. Research on the preparation of metallic magnesium and light-metal alloys, principally with aluminium, is under investigation by the Board of Scientific & Industrial Research.

*Manganese* — Annual exports from India of high-grade manganese have often touched the 1 million ton mark during the last 20 years. Proved reserves of the richer grade ores (48 to 70 per cent) are computed at only about 15 to 20 million tons; the reserves of lower-grade ores (Mn 40 to 30 per cent) are several times this magnitude. Beneficiation of the latter by simple mechanical means will greatly add to the country's resources in this metal. Manufacture of ferro-manganese in India is being investigated and the unrestricted exports of manganese ore has now been controlled by the Government.

*Nickel* — There is a paucity of nickel-ore in India; the only notable occurrence of nickel-ores is reported from a locality in Nepal, but detailed prospecting and geological examination have not been done to ascertain the quantity and extent of the deposit.

The Bawdwin Mines of Burma produced annually nickel speiss averaging 3,000 tons, during pre-war years, containing 25 to 30 per cent of nickel. Annual consumption of nickel in India is about 2,000 tons.

*Strontium* — Large deposits of remarkably pure celestite, aggregating to about a million tons each, occur in the Trichinopoly district of Madras and in a West Punjab district (Pakistan). The mineral has not found industrial use in India yet.

*Thorium* — The thorium resources of India are considerable.  $\text{ThO}_2$  is a constant ingredient of the mineral monazite occurring in the form of beach sands, covering large areas of the Travancore coast. These beach deposits constitute a large potential source of thorium, which has now assumed strategic importance as an atomic fuel or a source of atomic energy. The export of monazite, for strategic reasons, is now prohibited by Government.

*Titanium* — The mineral ilmenite is widely distributed in India and constitutes an important mineral asset of the country. It occurs as highly concentrated black sand along a hundred-mile stretch of the Travancore coast. About 75 per cent of the world's requirements in titanium, ranging between 200,000 to 300,000 tons per annum were, prior to 1940, supplied by Travancore. The percentage of  $\text{TiO}_2$  in Travancore ilmenite runs to 52 to 62; the total reserves of ilmenite are estimated at over 300 million tons. Rutile is associated with ilmenite sands.

A considerable accumulation of ilmenite occurs in association with the alluvial deposits of tin-ore in Malaya from which it is separated by magnetic concentration.

*Tin* — This metal does not occur in India in any appreciable quantity. Malaya is the world's largest supplier of tin-ore, the quantity varying from 33,000 to 77,000 tons per annum (pre-war production), being about one-third the world's basic tonnage in this metal. This figure represents the entire output of Malayan mines. During the last 20 years, many of the richer placers have been exhausted. The largest proportion of the ore is won by dredging the alluvial and eluvial deposits. The remaining ore reserves are considered to be large. Indonesia and Siam are the next largest producers of tin-ore in south-east Asia, their share being 19,000 and 10,500 tons respectively per annum. China comes next with 6,500 tons. All the ore is mined by more or less primitive methods from shallow alluvial flats.

*Tungsten* — A small deposit of this metal was worked in India during war years. The world's largest producer of this valuable alloy metal is China which has yet maintained its lead in this metal in spite of her disturbed political conditions. The production in pre-war years was 11,000 to 17,000 tons, the present output being much smaller. The ore is worked from large deposits of detrital stream-tin. The next largest exporter is Burma with 5,000 to 6,000 tons (pre-war) followed by Chosen, 1,500 to 2,000 tons, and Malaya, from a few hundred to 2,000 tons. No part of

the ore won is used indigenously in these countries.

*Uranium* — Sporadic deposits of uranium-bearing minerals have been found in various localities in India, the largest being segregations of pitch-blende in mica-pegmatites in Bihar, from which a few tons were obtained some years ago. Geological work has revealed that such deposits are incapable of supporting continuous mining. Minerals containing the rare earths and metals have been observed in pegmatites traversing the mica fields of Bihar and Madras, viz. samarskite, gadolinite, columbite, tantalite, allanite, triplite, torbernite and thorianite.

*Vanadium* — Considerable reserves of this metal have been localized in parts of Bihar and Orissa as vanadium-bearing iron-ores; the vanadium occurs in these areas in amounts varying from 0.8 to 3 per cent. The total reserves of vanadiferous iron-ore is estimated at 3 to 5 million tons. The utilization of these ores for manufacture of vanadium-steel is under consideration.

*Zinc* — Zinc lodes have been observed at a few localities in India but no smelting of zinc is practised in India today. Slag deposits of large size in several districts suggest a flourishing zinc industry in medieval times, the zinc being refined for manufacture of brass and bronze for local consumption. The annual consumption of zinc in India, at present about 30,000 to 35,000 tons, is wholly met from imports.

*Zirconium* — Reserves of this metal are ample, but at present a fitful output of only 100 to 1,500 tons of the mineral zircon is worked for purposes of export.



# Characteristics of the Ionosphere over Calcutta (July 1949)

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THE following are the ionospheric data observed at Calcutta for the month of July 1949.

Fig. 1 represents the mean hourly values of the penetration frequencies of the E and  $E_s$  regions and the penetration frequencies and virtual heights of region  $F_2$ . The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the prediction of the maximum usable frequencies which can be used for different distances of transmission by reflection at the F region over Calcutta for the month of October 1949. Table I gives the different occasions during routine observation when sporadic E ionization was observed and the values of the corresponding penetration frequencies and heights.

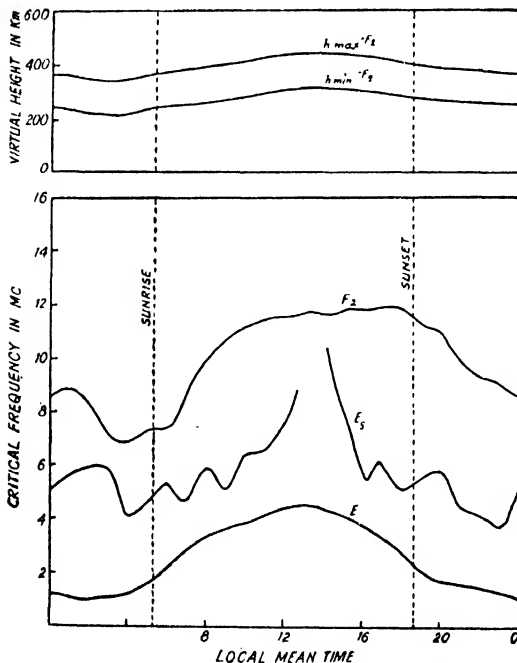
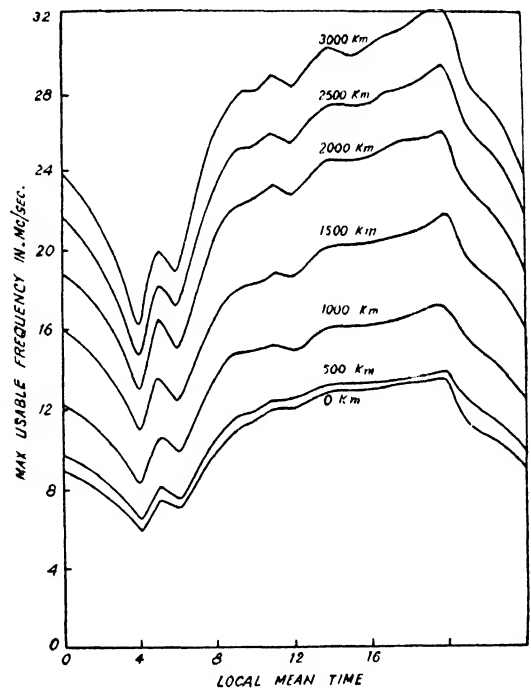


FIG. 1 — JULY 1949.

Sporadic E ionization was found to occur frequently during early morning hours but less in the afternoon unlike the previous month. Absorption of normal E echoes at mid-day was less than in the previous month. The behaviour of region  $F_2$  was found to be normal in this month.



AT POINT OF REFLECTION.  
FIG. 2 — PREDICTED M.U.F. VIA  $F_2$  LAYER DURING THE MONTH OF OCTOBER 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f^oE_s$	$hE_s$
			Mc.	Km.
July 1949	1	05.00	2.70	105
		08.00	7.45	120
		12.00	5.40	120
		14.00	10.00	135
		16.00	6.00	120
		17.00	4.25	120
		18.00	7.00	120
		20.00	3.25	105
		21.00	3.00	105
		22.00	3.95	90

TABLE I -- contd.

MONTH & YEAR	DATE	Hour	$f^{\circ}E_s$ Mc.	$hE_s$ Km.
July 1949	2	00.00	2.80	90
		10.00	3.00	120
		11.00	6.20	120
	4	18.00	3.30	120
		19.00	4.00	120
		20.00	4.75	120
		23.00	4.05	105
	5	00.00	4.25	105
		01.00	3.90	105
		06.00	5.00	120
		07.00	5.20	120
		08.00	6.10	135
		10.00	6.50	135
		21.00	3.50	135
		22.00	3.00	120
		23.00	2.50	105
	6	05.00	5.75	120
		06.00	8.20	135
		07.00	4.70	135
		09.00	5.00	135
		10.00	6.15	135
		17.00	5.30	135
		18.00	5.40	120
	7	08.00	7.00	120
		09.00	5.50	120
		16.00	4.25	120
		17.00	4.70	120
		21.00	2.30	105
		22.00	2.50	105
		23.00	2.00	90
	8	07.00	4.25	105
		10.00	4.50	105
		16.00	6.10	120
		20.00	6.00	120
		21.00	4.25	120
	9	06.00	6.00	120
		07.00	4.25	120
		09.00	5.00	120
		12.00	4.70	120
	11	16.00	4.70	120
		17.00	10.40	135
		18.00	7.85	135
		19.00	7.00	120
		21.00	6.75	120
		22.00	3.25	105
		23.00	4.70	105
	12	00.00	7.95	105
		01.00	7.95	105
		02.00	7.95	105
		03.00	6.10	105
		04.00	4.70	105
		07.00	4.70	120
		08.00	5.35	120
		09.00	6.00	135
		11.00	6.00	135
		19.00	4.20	120
		20.00	4.00	120
		21.00	4.25	105
		22.00	4.20	105
		23.00	4.00	105
	13	00.00	4.00	90
		02.00	5.30	90
		06.00	5.20	120
		09.00	4.20	120
		18.00	5.30	120

TABLE I -- contd.

MONTH & YEAR	DATE	Hour	$f^{\circ}E_s$ Mc.	$hE_s$ Km.
July 1949	14	08.00	4.60	105
		09.00	5.00	120
		10.00	4.90	120
		23.00	2.70	90
	15	01.00	6.65	90
		02.00	6.00	90
		03.00	5.50	90
		04.00	4.95	90
		05.00	4.95	105
		07.00	4.95	105
		09.00	5.40	105
	19	22.00	4.60	90
		23.00	3.20	90
	20	00.00	3.00	90
		01.00	3.20	90
		02.00	4.00	90
		06.00	5.00	105
		07.00	4.80	105
	21	10.00	4.70	120
	22	04.00	2.50	90
		05.00	2.50	90
		06.00	2.50	105
		07.00	4.65	105
		08.00	4.75	105
		09.00	4.75	120
		16.00	4.60	120
	23	17.00	4.25	120
	25	10.00	4.70	120
		11.00	4.80	120
	26	18.00	4.25	120
		19.00	4.00	120
	27	08.00	4.55	120
		10.00	11.25	135
		12.00	12.50	135
		15.00	7.20	120
		16.00	7.00	120
		17.00	4.70	120
		18.00	3.85	120
		19.00	11.75	135
		20.00	11.00	135
		21.00	10.80	135
		22.00	8.00	120
		23.00	6.70	120
		00.00	8.50	105
	28	01.00	6.90	105
		09.00	4.50	105
		10.00	11.25	120
		11.00	10.00	135
		13.00	13.10	150
		14.00	10.80	150
		17.00	9.60	135
	29	08.00	8.20	120
		11.00	6.00	120
		12.00	7.50	135
		17.00	3.40	120
		18.00	3.90	120
	30	09.00	5.25	120
		10.00	6.00	135
		14.00	12.00	150
		18.00	4.70	135
		19.00	3.80	120
		01.00	4.50	105
		05.00	7.00	120
		07.00	4.50	120
		09.00	5.25	120

# Atomic Nuclear Models & Beta Decay

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## 1. Radioactivity & Nuclear

### Model for the Atom

**R**ADIOACTIVITY, which is characterized by spontaneous emission of unique radiations, was discovered by Becquerel in 1896. Soon after the discovery of the radioactivity of uranium, a number of other heavy elements were also found to be radioactive. An interpretation of this phenomenon was made on the basis of Rutherford nuclear model for the atom. The atom consists of a central heavy particle or nucleus in which most of the mass and the total positive charge are concentrated. The nucleus is surrounded by a certain number of moving electrons in configuration whose total negative charge compensates the nuclear charge giving rise to neutral atom. The charge of the nucleus is thus an integral multiple  $Z$  of the electronic charge.

Chemical and spectroscopic properties are determined by the nuclear charge which determines the number of electrons and their distribution in the extra nuclear electronic configuration of the atom while mass properties exhibited by the atom are due to the nuclear structure, the mass being given by the mass of the nucleus. Thus, a clear and distinct separation of the chemical and spectroscopic properties from the mass properties are provided by the nucleus. Hence there can be, as there actually are, isotopic atoms, that is, atoms of different masses having the same chemical properties and practically the same spectrum.

The phenomenon of radioactivity is due to instability in the very structure of the nucleus of the atom which leads to a change in either the mass and charge properties together or in only the charge properties with the mass properties practically unaffected. In other words, the spontaneous emission of radiations gives rise to a change in either the mass and charge properties jointly or in only the charge properties. These radiations emitted by radioactive elements, as a result of careful investigation by

absorption and other methods, are found to be of three different and clearly defined groups, alpha, beta and gamma.

Of these, the alpha rays are found to be doubly charged atoms of helium. In other words, they are particles with a positive charge of two units and a mass of four atomic mass units,  ${}_2\text{He}^4$ . They, as positively charged particles, are deflected by magnetic and electric fields. The beta rays are positively or negatively charged, high-speed positrons or electrons and, as such, are also deflected by these fields. The gamma rays, on the other hand, are electro-magnetic radiations of very high frequency and they are unaffected by magnetic and electric fields.

## 2. The Proton-electron Nuclear

### Model & Emission of Beta Rays

The emission of radiations accompanied by a change of mass properties indicates that the atomic nucleus is not a simple particle, rather it is composite, and properties of nuclei such as momentum or spin which can be deduced from optical spectra, confirm the assumption that this composite consists of elementary particles. In the earliest development of the nuclear theory, the nucleus was assumed to be built up of protons and electrons. The proton-electron nuclear model encountered many difficulties.

The emission of electrons as beta rays from radioactive nuclei was long regarded as direct evidence for the existence of electrons in nuclei. With the discovery of numerous artificially produced radioactive nuclei which emit positrons instead of electrons in their energy spectra, this argument proved to be untenable, since beta radioactive substances may emit either positive or negative electrons from their nuclei.

The electron and proton, by direct measurement, are each found to have a spin of  $\frac{1}{2}$ . Hence the angular momentum of a nucleus, composed of an odd total number of particles, must be half integral. Band



spectra show that the nuclear spin of  ${}^7\text{N}^{14}$  is 1. Therefore, it cannot contain an odd number of particles, 14 protons and 7 electrons. Similarly  ${}^1\text{H}^2$ ,  ${}^3\text{Li}^6$  and  ${}^5\text{B}^{10}$ , each with spin 1, cannot contain odd number of particles as required by nuclear proton-electron hypothesis.

${}^7\text{N}^{14}$ , as shown by band spectra, follows Bose-Einstein statistics and hence must contain an even number of nuclear particles. If the nitrogen nucleus contained 14 protons and 7 electrons or 21 nuclear particles in all, it would have to obey Fermi-Dirac statistics, since the total number of particles is odd. Also  ${}^1\text{H}^2$ ,  ${}^3\text{Li}^6$  and  ${}^5\text{B}^{10}$  which would have to follow Fermi-Dirac statistics actually follow Bose-Einstein statistics.

Thus the spin and statistics properties, which the proton-electron nuclear model is expected to have, come into conflict with those actually observed in a nucleus.

Measured nuclear magnetic dipole moments are all of the order of the nuclear magneton  $e\hbar/2MC$ . The Bohr magneton containing the electron mass  $m$ , instead of the proton mass  $M$ , is  $M/m$  which is equal to 1,840 times as large as the nuclear magneton. The measured magnetic dipole moment of the electron is one Bohr magneton and is negative in sign, corresponding to the rotation of a negative charge in the spin direction. The presence of electrons in the nucleus would, therefore, require nuclear magnetic moments nearly 2,000 times greater than what is actually observed and these would be negative in sign.

If the beta particles emitted by radioactive elements, as the proton-electron hypothesis implies, be high-energy constituents of their nuclei, which have mean life from incredibly small fraction of a second up to a million years or more, then there must be some power to hold these electrons for this length of time inside the nuclei. In analogy to alpha particles, one may attempt to devise a potential barrier. But it is impossible, as Bethe and Bacher<sup>1</sup> have shown, to devise a nuclear potential barrier to hold electrons inside the nucleus unless the height of the barrier is of the order of 13 times the height of the  $k$ -level in heavy atoms. Such a nuclear potential would cause large perturbations of the optical and X-ray levels which are definitely not observed.

The electrons observed in beta disintegration cannot exist in the nucleus prior

to their emission as beta rays. This inability can be judged quantitatively from the fundamentals of wave mechanics. The De Broglie<sup>2</sup> wavelength  $\lambda$  associated with a particle of momentum,  $P=mv$ , is given by

$$\lambda = \frac{h}{p} \quad (1)$$

where  $h$  is Planck's constant. Now, to derive the relativistic expression for momentum  $P$  from equation for kinetic energy, let  $C$  be the velocity of light,  $m$  the mass of the particle moving with a velocity  $v$ ,  $m_0$  its rest mass and  $E$  the kinetic energy of motion, given by

$$\begin{aligned} E &= m_0 C^2 \left( \frac{1}{\sqrt{1 - \frac{v^2}{c^2}}} - 1 \right) \\ &= (mC^2 - m_0 C^2) \\ &= \Delta m C^2 \end{aligned}$$

Then the momentum, expressed in terms of the kinetic energy, has the form

$$P = mv = \frac{1}{C} \sqrt{2Em_0 C^2 + E^2} \quad (2)$$

Beta rays from most of the naturally radioactive elements have energies of 1.5 MEV or less, and an electron of this energy whose wavelength  $\lambda$ , from equations 1 and 2, is  $630 \times 10^{-13}$  cm. could not exist as a standing wave even in the heaviest nucleus of uranium since its diameter is  $25 \times 10^{-13}$  cm.

This fact becomes more obvious when the quantization of moment of momentum of an electron is considered. Heisenberg<sup>3</sup> has shown that this quantization implies that simultaneous knowledge of the position and momentum of a particle cannot have an accuracy greater than that implied in the expression

$$\Delta P \times \Delta q = \frac{h}{2\pi} \quad (3)$$

where  $\Delta P$  and  $\Delta q$  are simultaneous indeterminacies in momentum and position. To consider minimum indeterminacy of the position of an electron, a maximum indeterminacy in its momentum may be assumed. Let this indeterminacy be its entire momentum  $P$ . Then the minimum indeterminacy in position is

$$\Delta q_{\min.} = \frac{h}{2\pi P} = \frac{\lambda}{2\pi} \quad (4)$$

i.e. the minimum indeterminacy is about De Broglie wavelength  $\lambda$  divided by  $2\pi$ .

Therefore, the minimum indeterminacy in position of an electron with an energy of 1.5 MEV is about  $100 \times 10^{-13}$  cm., or 4 times the diameter of the largest nucleus. Hence it could not be stated that the beta particle had ever existed in the nucleus.

From the considerations of the principle of indeterminacy, it can be found that if the position of an electron is to be known within  $\Delta q = 10 \times 10^{-13}$  cm. or about the diameter of the nitrogen nucleus, the energy of the electron must be of the order of 19 MEV. The total binding energy, however, of the  ${}^7\text{N}^{14}$  nucleus is only about 100 MEV, and if this were to be shared between 14 protons and 7 electrons in this nucleus, as the proton-electron nuclear model implies, then there would be too little energy per particle to meet the minimum energy requirement and, therefore, these electrons could not be confined to the nuclear volume.

Thus, these considerations show that the proton-electron nuclear model is untenable and that the fundamental difficulties associated with this hypothesis can be surmounted when the electron is, as proposed by Heisenberg and Majorana, replaced by a heavy particle without charge.

### 3. The Neutron-proton Nuclear Model & Beta Decay

With the break down of the proton-electron hypothesis, there came the neutron-proton nuclear model with the proof of the existence of another fundamental nuclear particle, the neutron. Chadwick's discovery of neutron<sup>4,5</sup> which threw great light on the structure of atomic nuclei, confirmed the hypothesis of a nucleus composed of neutrons (neutral particles) and protons (hydrogen nuclei). According to this hypothesis, the atomic number  $Z$  of an element is the number of protons in its nucleus while its mass number  $A$  is the total number of protons and neutrons in the nucleus, or

$$Z = N_p, \text{ and } A = (N_p + N_n) \quad (5)$$

where  $N_p$  and  $N_n$  are the number of protons and neutrons respectively.

The neutron-proton nuclear model, consisting of heavy particles, surmounts some of the principal difficulties which the former encountered. On this hypothesis, since neutron, like proton, follows the Fermi statistics, it is expected that nuclei of odd mass number obey Fermi-Dirac statistics while those of even mass number obey Bose-

Einstein statistics. Also, since the spin of either one of the elementary nuclear constituents is half integral, the nuclear spin is expected to be half integral or integral according to whether the mass number is odd or even. The nuclear spin and statistics properties for stationary states, expected on this neutron-proton hypothesis, have been actually verified by experiments. Further, the nuclear magnetic dipole moments which, on this hypothesis, are all expected to be of the same order of magnitude as those of the proton and neutron, are also confirmed by experimental evidence.

In discussing the proton-electron nuclear hypothesis, it has been shown that the presence of an electron in a nucleus is an objectionable feature both from the point of view of the nuclear dynamical and statistical properties as well as from that of beta decay. If the presence of electrons in the nuclear structure be excluded, then it is natural to suppose that in the process of beta decay, the electrons are formed at the moment of their actual emission in a manner analogous to the emission of light quanta from atoms. This assumption solves the difficulties of the nuclear magnetic moments, the magnitude of wave function in relation to the nuclear dimensions and assumption of potential barrier encountered by the proton-electron nuclear model. There, however, remain the difficulties of statistics and spin. In addition to these, a still more serious difficulty is that beta disintegration electrons have energies distributed over a wide range from zero to a fairly defined maximum  $E_0$  in spite of the fact that the initial nuclei, before the emission of beta rays and, after their emission, the residual product nuclei have quite exactly determined energies.

Thus the situation to be confronted with is that the parent nucleus, in a quantized state of definite energy, emits an electron leaving a product nucleus also in a quantized state of definite energy. The emitted electron, however, does not carry away the energy difference  $\Delta E$  between these two quantized states but any energy between this value and the energy value which corresponds to the rest mass of an electron, the exact energy in each individual case being apparently determined by chance. To account for this, Bohr proposed the theory of non-conservation of energy in the process of beta disintegration.

This hypothesis, however, is in conflict with the observation that the total energy is conserved in the process of beta emission as far as the upper limits of beta-ray spectra are concerned. It encounters also other serious theoretical and experimental difficulties.

Then Pauli pointed out that the principle of conservation of energy is valid and the conservation laws may be retained if it be assumed that in beta disintegration a new particle which is escaping observation is emitted along with the beta particle, carrying away the missing energy, momentum and spin. According to Pauli, this new kind of particle which he calls neutrino is devoid of electric charge and is of very small mass. It has practically no interaction with matter.

In explaining the facts of continuous energy spectra, Pauli assumes that in each beta decay, an electron and a neutrino are simultaneously emitted, that the maximum energy of beta rays occurs when the neutrino is emitted with zero velocity and that the energy difference  $\Delta E$  between the initial and the product nuclei is distributed among the electron and the neutrino so that the electron receives a part of  $\Delta E$ , varying from case to case. Then the maximum kinetic energy, which the electron may receive, is given by

$$E_0 = \Delta E - (m_e + m_\nu)C^2 \quad (6)$$

where  $m_e$  and  $m_\nu$  are the rest masses of the electron and neutrino respectively and  $E_0$  is the disintegration energy, represented by the upper limit of the energy spectrum.

The difficulties of spin and statistics for a stationary state of a nucleus are, on the basis of neutron-proton nuclear model, overcome, but we are confronted with these difficulties for nuclear instability state with beta emission. Since in the beta transformation the atomic weight of the nucleus does not change, it is expected that the properties of a nucleus having an integral or half integral spin and Fermi or Bose statistics would be reversed by the emission of an electron—a particle with half spin and Fermi statistics. These properties, however, are not changed by this transformation of initial nucleus into product nucleus. The unchangeability of these properties can be accounted on the assumption of the emission of the second particle, the neutrino of half spin and Fermi statistics, inasmuch as the resultant spin of the

simultaneously emitted electron and neutrino is integral (1 or 0) and the neutrino and electron, both being elementary particles, each with Fermi statistics, the initial statistics properties of the parent nucleus are retained by the product nucleus.

From the considerations outlined above and from the results of experience, it may be stated that a neutrino has no charge and has a very small mass, only a fraction of the electron rest mass. It follows Fermi statistics and its spin  $\frac{1}{2}\hbar = \frac{h}{4\pi}$  where  $h$  is

Planck's constant. Its magnetic moment, if any, must be less than  $7.0 \times 10^{-6}$  magneton. Nahmias' search<sup>6</sup> for ionization by neutrino, using strong radioactive sources shielded by large amounts of lead (about 1 metre lead) in order to absorb alpha, beta and gamma rays and leave only the neutrinos, shows that neutrinos cannot form more than 1 ion in about 500,000 kilometres path in air, indicating that their magnetic moment, if any, must be smaller than  $7.0 \times 10^{-6}$  magneton. It has no detectable effects in the free state except the momentum recoil of the initial nucleus. The data of the automatic Wilson cloud chamber experiments with  $^{38}_{17}\text{Cl}$ , conducted by Crane and Halpern<sup>7,8</sup>, and Allen<sup>9</sup>, show that the momentum of the recoil nucleus is greater than what it could have received from beta ray alone, thus indicating the existence of a third particle taking part in the disintegration. So the momentum of the recoil nucleus, over and above what it could have received from the beta particle, must be considered to have received from the third particle, the neutrino.

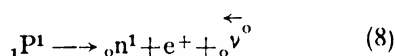
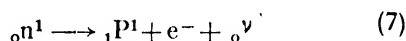
#### 4. Beta Decay & the Theory of Allowed Transitions

The phenomenon of beta emission is in many respects analogous to the phenomenon of emission of a light quantum in the transition of an electrically charged particle from a higher to a lower quantum state. A photon is produced in the quantum transition effected by the coupling of the electron in atom. Similarly, an electron (negative or positive) and neutrino or antineutrino are formed by the transition of heavy nuclear particle (nucleon) caused by the coupling of the nucleons with the electron-neutrino field.

Hence, neutrinos or antineutrinos may obey a wave equation similar to Dirac equation, the charge being zero and mass negligible. The solutions of this equation

give values with positive as well as negative energy. All states of negative energy must be assumed to be ordinarily filled in order to avoid difficulties associated with the possibility of transitions from positive to negative energy. In the case of an electron, an empty negative energy state is equivalent to a particle analogous to positron, while in the case of neutrino, the empty negative energy state is equivalent to a particle analogous to antineutrino. Thus a neutrino is emitted along with the electron and an antineutrino along with the positron. Antineutrino has no property such as charge or magnetic moment that distinguishes it from the neutrino except for the difference in connection with positron and electron emissions and the associated theoretical values.

In beta disintegrations, the nuclear charge is changed by one unit while the mass number remains the same. So the phenomenon of beta disintegration can be considered as the transition of the nuclear heavy particle (nucleon) from neutron eigen state to the proton eigen state in the case of the emission of electron and neutrino or from proton eigen state to the neutron eigen state in the case of the emission of positron and antineutrino which, when symbolically expressed, has the forms:



Following the lines of analogy outlined above, Fermi<sup>10</sup> developed a theory of beta disintegration on the basis of an analysis of interaction between neutrons and protons, and electrons and neutrinos. The theory calculates the disintegration probability and predicts the shape of the continuous spectrum in which the two light particles, electron and neutrino or positron and antineutrino, share the available energy.

The calculations of the theory are rendered easier by simplifying assumptions in which the wave functions of light particles are considered as constant over the whole nucleus and equal to their value at the nuclear radius  $R_0$ , the velocity of the heavy particles is assumed as negligible in comparison with the velocity of light, and the forces of interaction are treated as those of short ranges and, therefore, the interaction as one depending mainly on the values of the wave functions in the neighbourhood of nucleons.

Now, to proceed into the actual working of the theory of allowed transitions, consider two light particles, an electron and a neutrino or a positron and an antineutrino. Let the wave function of the electron be denoted by  $\phi$  and that of neutrino by  $\psi$  and  $X$  be co-ordinates of both particles. Then the interaction will be, in general, a function of

$$\phi(x) \frac{d}{dt} \phi(x) \frac{d^2}{dt^2} \phi(x), \text{ etc., and}$$

$$\psi(x) \frac{d}{dt} \psi(x) \frac{d^2}{dt^2} \psi(x), \text{ etc.} \quad (9)$$

As the simplest hypothesis, the interaction may be assumed as one which depends on the wave functions of the light particles but not on their derivatives and, on this assumption, the Hamiltonian of this interaction may be expressed in the form:

$$H \sim g \int \Phi^*(x) \Psi(x) \phi^*(x) \psi(x) \quad (10)$$

where  $g$  is the constant of coupling between the nucleons and electron-neutrino field and  $\Phi$  and  $\Psi$  are the wave functions of proton and neutron respectively.

The forms of experimentally obtained continuous energy spectra are found to have a better fit with the calculated distribution curves if the mass of neutrino be assumed to be vanishingly small, i.e. of the order of zero compared with the mass of an electron. Under the simplifying assumptions mentioned and the condition that  $\mu \sim 0$ , the transition probability  $P_{(E)}$  in which an electron is emitted with an energy between  $E$  and  $E+dE$  is given by

$$P_{(E)} dE = G^2 |\chi|^2 E \sqrt{E^2 - 1} (E_0 - E)^2 dE \quad (11)$$

where  $E_0$  is the total available energy in units of  $MC^2$ ,

$$\chi = \int \Phi \Psi dW,$$

$\Phi$  and  $\Psi$  being the wave functions for proton and neutron respectively and the integration being a summation over all possible spins and directions of the nucleons in the field of nuclear forces,

$$G = g \frac{m^2 C}{\sqrt{2\pi} \hbar^3}$$

is the universal constant indicating the forces of interaction and

$$E \sqrt{E^2 - 1} (E_0 - E)^2 dE$$

is the statistical factor of the transition probability. The expression (11) is a good approximation for  $Z \sim O$ . In heavier elements, the influence of Coulomb field is appreciable

and for these elements the allowed transition probability expression (11), however, will be of the form :

$$P_{(E)}dE = G^2 |\chi|^2 F_{(Z,E)} E \sqrt{E^2 - 1} \times [E_0 - E]^2 J E \quad (12)$$

where  $F_{(Z,E)}$  is a factor which determines the influence of Coulomb field on the emission of electrons. This factor is given by

$$F_{(Z,E)} = \frac{4}{[\Gamma(1+2S)]^2} \times (2\sqrt{E^2 - 1} R_0)^{2S-2} e^{\frac{\pi \alpha Z E}{\sqrt{E^2 - 1}}} \times \left[ \Gamma \left( S + i \frac{\alpha Z E}{\sqrt{E^2 - 1}} \right) \right]^2 \frac{1+S}{2} \quad (13)$$

where  $S = \sqrt{1 - \alpha^2 Z^2}$ , the fine structure constant  $\alpha = \frac{1}{137}$ ,  $R_0$  is the nuclear radius and  $\Gamma$  is gamma function. The allowed transition probability expression (12) did not agree with the available experimental data in that it did not predict as much electron density at low energies as an experimentally obtained spectrum actually indicates. The discrepancy between theory and experiment could be minimized if the theory would be modified so as to permit a higher probability for the emission of high energy neutrinos. Konopinski and Uhlenbeck<sup>11</sup> found a way to effect this modification by showing that although Fermi had made the most plausible assumption concerning the interaction of electron and neutrino with the nucleus, other assumptions were nevertheless possible which could lead to different probability expressions. They worked the theory for several different types of interaction and found that one, in particular, in which not only the eigen functions of electron and neutrino but also their derivatives with respect to co-ordinates were introduced into the expression for interaction, led to a modified form of probability expression. This new expression which seemed to have agreed with the available experimental data, differed from that of Fermi in that the factor  $(E_0 - E)^4$  appeared in the place of  $(E_0 - E)^2$ . The fourth power of neutrino as given by Konopinski-Uhlenbeck's expression instead of the second power as in (12) increases the probability of fast neutrinos and slow electrons and shifts the most probable electron energy approximately to  $E_0/3$  for large  $E_0$ .

From the considerations outlined above, it is obvious that the energy spectrum of electrons is given by

$$N_{(E)}dE \sim E \sqrt{E^2 - 1} (E_0 - E)^2 dE \quad (14)$$

$$\text{and } N_{(E)}dE \sim E \sqrt{E^2 - 1} (E_0 - E)^4 dE \quad (15)$$

in accordance with the Fermi allowed transition probability and the Konopinski-Uhlenbeck modification respectively. In these expressions  $N(E)dE$  are relative numbers of particles in equal intervals of energy. From the allowed transition probability expression (12) and the energy spectrum expressions (14) and (15), it follows that a plot

$$\left[ \frac{N_{(E)} dE}{P^2 F_{(Z,E)}} \right]^{\frac{1}{k}}$$

against energy  $E$  should yield a straight line with its intercept with energy axis representing the maximum available energy  $E_0$ , if the Fermi theory ( $K=2$ ) or Konopinski's theory ( $K=4$ ) is correct.

Early experimental data of certain continuous spectra appeared to satisfy Konopinski-Uhlenbeck allowed transition probability equation better than that of Fermi. But results of subsequent measurements carefully carried out with improved experimental technique had shown that Konopinski-Uhlenbeck modification was instigated by the influence of scattering of electrons caused by the thickness of the source and improper source mounting technique.

## 5. Allowed & Forbidden Transitions

Owing to the simplifying assumptions already dealt with, the matrix element  $\chi = \int \Phi \Psi dw$  is produced in the disintegration probability expression (12). This element will be near unity if the wave functions of the nucleons in the initial and final states are alike or very much alike. If  $\chi \sim 1$  or  $\chi \neq 0$ , the spin change at the disintegration  $\Delta I = 0$ . This condition constitutes the Fermi selection rule for the allowed transition probability, the theory of which is briefly outlined in the last section (section 4).

If the matrix element  $\chi$  vanishes for the transition ( $\chi = 0$ ) which occurs most usually with the change of total angular momentum, the transition will be forbidden. The forbidden transitions of the first, second and third orders are distinguished according to changes of  $I$  by  $\Delta I = 1, 2, 3 \dots$ . These forbidden transitions are comparatively much less probable than the allowed transitions with  $\Delta I = 0$ . The probability of forbidden

transitions of higher order decreases by a factor of approximately

$$\frac{R_0}{\hbar/mc} = \frac{4 \times 10^{-13}}{3.86 \times 10^{-11}} \sim \frac{1}{100}$$

per order. A forbidden transition is characterized by relatively long life, and by an energy distribution curve other than that for the allowed Fermi distribution. Fermi made approximate calculation and this calculation indicates that the energy distribution curve at low energies should be at a position beneath the allowed Fermi curve in the case of forbidden transitions. This indication, however, could not be reconciled with some of the available experimental data<sup>12</sup>.

### 6. Life-time & Beta-disintegration Energy

The theory of beta decay indicates that there is a relation between life-time  $\tau$  for beta-ray emission and maximum disintegration energy  $E_0$  available for electron and neutrino, since the reciprocal of the life-time is the total transition probability. This quantity may, therefore, be derived from the transition probability which is obtained by integrating the energy distribution equation (10) thus:

$$\frac{1}{\tau} = G^2 |\chi|^2 f_{(Z, E_0)} \quad (16)$$

where  $G$  is a constant denoting the strength of interaction between the heavy particles and light particles,  $\chi$  is the matrix element for the transition and  $f_{(Z, E_0)}$  is the integral of the energy distribution given by

$$f_{(Z, E_0)} = \int_1^{E_0} F_{(Z, E)} E \sqrt{E^2 - 1} (E_0 - E)^2 dE \quad (17)$$

$E_0$  being in units of electron rest mass.

The reciprocal of the life-time  $\frac{1}{\tau}$  for beta emission is identical with beta disintegration constant  $\lambda$ .

If  $F_{(Z, E)} = 1$ , and this is certainly valid for all light elements, the disintegration constant  $\lambda$  may be expressed in this form:

$$\lambda = G^2 |\chi|^2 \int_1^{E_0} E \sqrt{E^2 - 1} (E_0 - E)^2 dE \quad (18)$$

From the expressions (16) and (17) it is obvious that  $f_{(Z, E_0)}$  varies rapidly with  $E_0$ , and thus the life-time  $\tau$  of the beta-disintegrating element decreases rapidly with increasing kinetic energy of the particle, so that for small kinetic energy of the beta particles when  $E \sqrt{E^2 - 1} \sim E$

$$\frac{1}{\tau} = E^5 \quad (19)$$

and for large kinetic energy when

$$E \sqrt{E^2 - 1} \sim E^2 \quad \frac{1}{\tau} = E^7 \quad (19a)$$

while for much higher kinetic energies when

$$\frac{1}{\tau} = E^9 \quad (19b)$$

$\frac{1}{\tau}$  and  $\lambda$ , as indicated by (16), (17) and (18),

are identical. Therefore, from (9), (19a) and (19b),  $\lambda$  may be expressed in terms of beta-disintegration energy  $E_0$  by

$$\log \lambda = \log \frac{1}{\tau} = K \cdot \log E_0 \quad (19c)$$

where  $E_0$  is the beta-disintegration energy in units of  $MC^2$  obtained from the value of the upper end point of the energy distribution curve and  $K=5$  or  $7$ , etc., according to the range of energy and to the nature and order of transition. The group of beta-decay processes to which the constant  $K=5$  holds good is interpreted as corresponding to allowed transitions while the second and the third groups to which the constant  $K=7$  and  $K=9$  hold are interpreted as corresponding to forbidden transitions of the first and the second orders respectively. The classification of beta-decay processes into two or three groups, each group belonging to allowed or one of various forbidden transitions, has been made by Sargent<sup>13</sup> on purely empirical basis which will be dealt in section 7.

### 7. Sargent Diagrams & Classification of Beta-decaying Processes

The Fermi theory of beta decay, as shown in the previous two sections, 5 and 6, has led to a classification of the beta-decay processes into allowed and forbidden transitions, and the forbidden into different orders. This theory also implies the relation between beta-disintegration energy  $E_0$  and its decay constant  $\lambda$  and thus offers, so to speak, an interpretation to the well-known Sargent empirical rule which, in a modified form, is similar to the theoretically obtained relation expressed by (19c).

Sargent found that plots of the logarithms of half lives or of the disintegration constants versus logarithm of the maximum disintegration energies  $E_0$  of the beta-decaying isotopes such as those given in Appendix I give different orders of curves

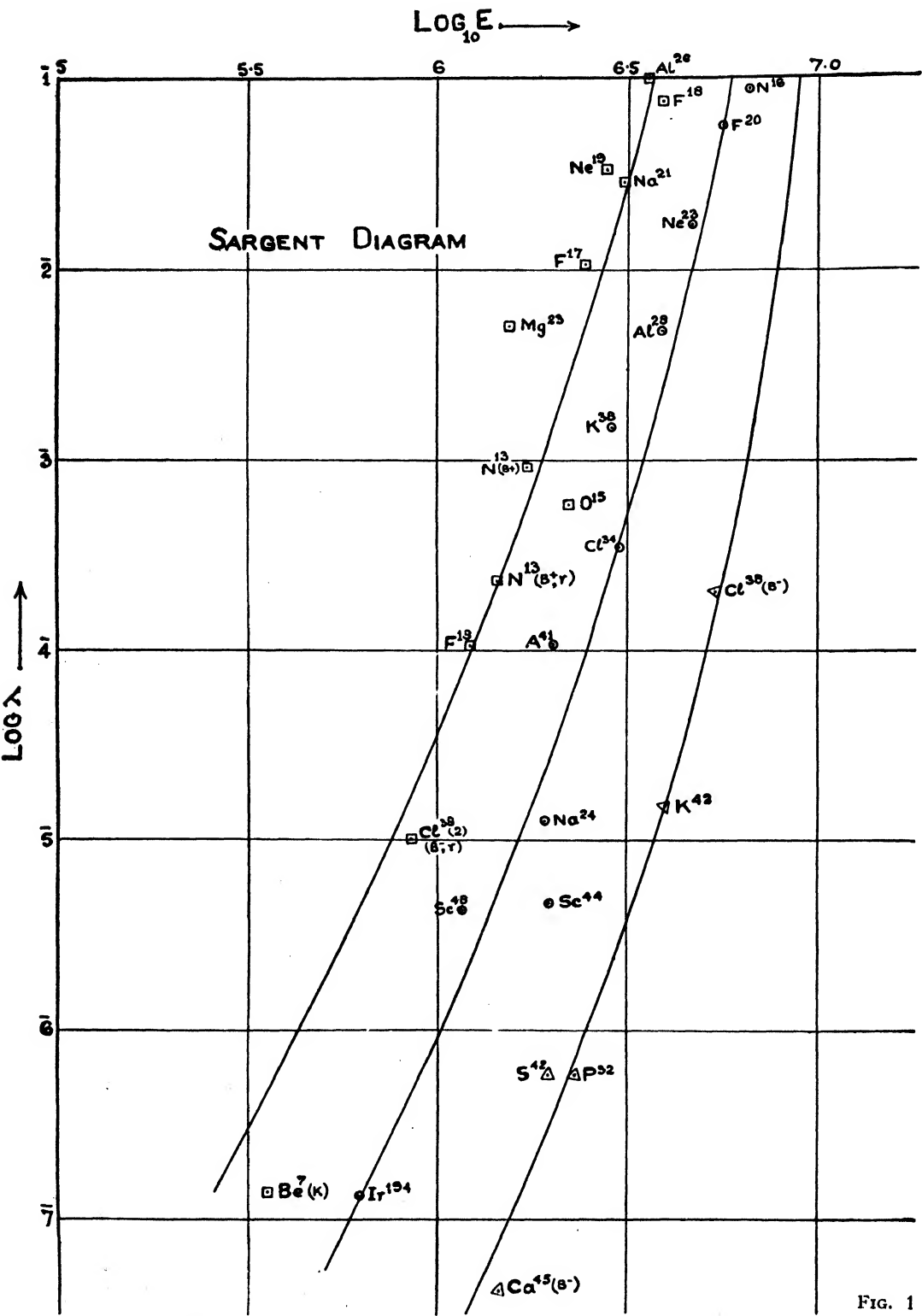


FIG. 1

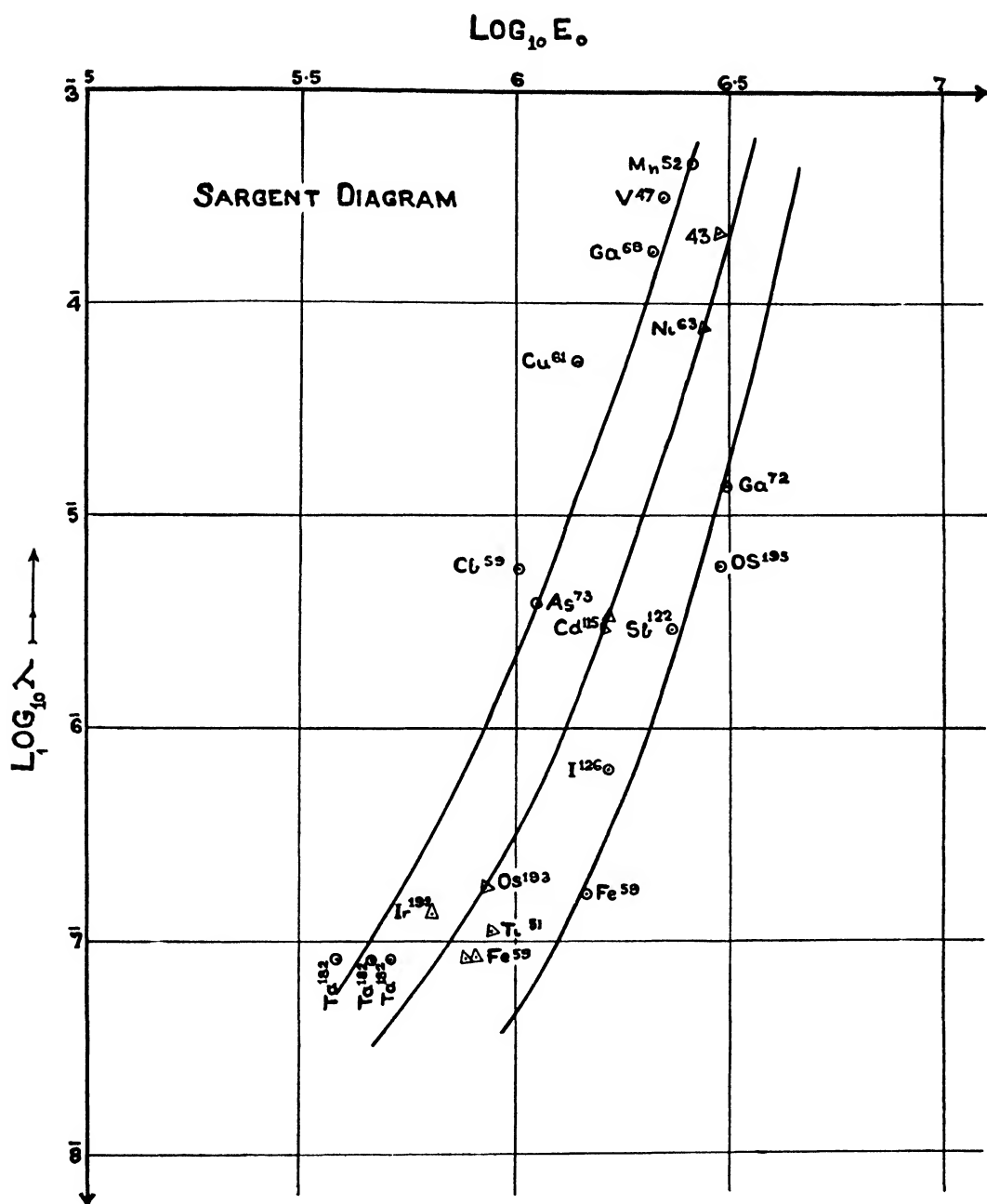


FIG. 2

as shown in Figs. 1, 2 and 3. In these three figures, the curves which represent the group of isotopes for which the constant  $K$  is a minimum, are curves of the group of isotopes of allowed transition while the other curves in each figure are of the groups of isotopes

of forbidden transitions. Thus, Sargent diagrams form an empirically obtained powerful graphical method of classifying the isotopes into allowed and forbidden transitions and those of forbidden transitions into different orders.



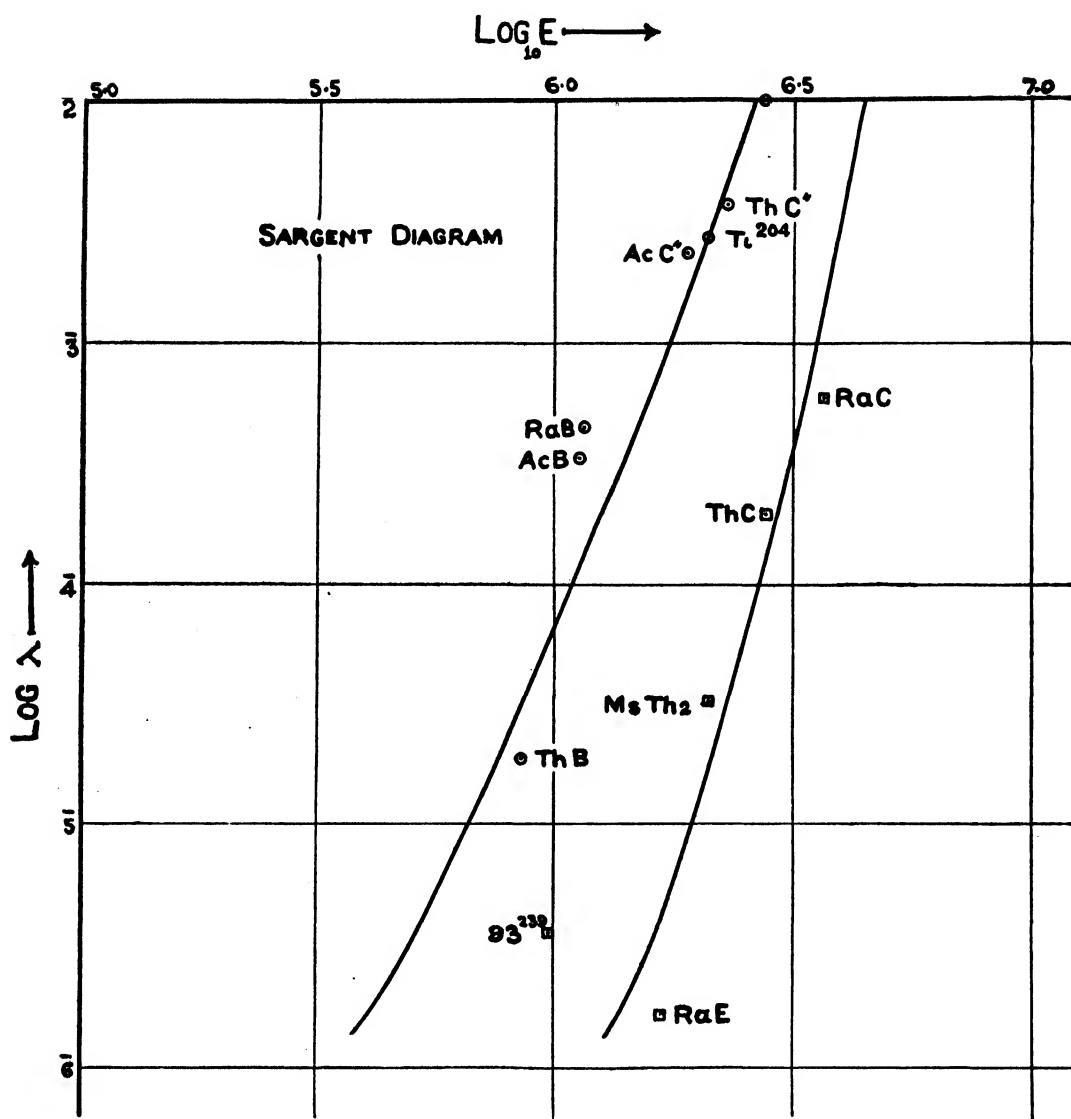


FIG. 3

### 8. Meson Theory & Beta Decay

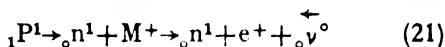
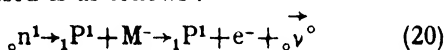
The matrix element  $\chi$  for allowed transition is of the order of unity and so the expression (18) for the disintegration constant  $\lambda$  readily gives the value of  $G$ . From the mean value so obtained, the coupling constant  $g$  may be found to be of the order of  $4 \times 10^{-50} \text{ cm.}^3 \text{ Erg}$ . The implication of this low value of the coupling constant is that Fermi field of forces would by far be too small to hold the nucleons together.

To surmount this difficulty, Yukawa<sup>14</sup> assumed that the force acting between

nucleons might be described by a meson field surrounding the one which acts on the other just as the Coulomb force between charged particles can be expressed in terms of the interaction of these particles with the electro-magnetic field. The quanta which surround a nucleon are mesons and if these are treated as having a finite mass  $m$ , the range of forces resulting from the meson field will be  $\hbar/mc$ , the Compton wavelength for the meson. From experiments on scattering of neutrons by protons, the range of forces is found to be of the order of  $2 \times 10^{-13} \text{ cm}$ .

With this range of forces, the meson rest mass should be of the order of 200 electron masses. Particles having this order of rest mass were discovered in cosmic rays. This discovery confirmed the required range of nuclear forces as produced by the Yukawa scalar meson theory describing the nature of the meson field.

The meson is ascribed an integral spin, and as assumed and subsequently confirmed by its discovery in cosmic rays, it is either negative or positive or neutral. The field, though virtual in its nature, may be described by the meson particle which has, so to say, its formation at a beta disintegration as an intermediary between this disintegration and the emission of light particles. The meson is capable of exchange between a neutron and proton or vice versa between a proton and neutron or any pair of nuclear heavy particles depending upon whether the meson is negative or positive or neutral. It has a very short half-life period of the order of a micro-second and disintegrates into light particles, negatron and neutrino or positron and antineutrino. Thus the interaction symbolically expressed is as follows :



where  ${}_0n^1$ ,  ${}_1P^1$ ,  $M^-$ ,  $M^+$ ,  $e^-$ ,  $e^+$ ,  ${}_0\nu^0$  and  ${}_0\bar{\nu}^0$  are neutron, proton, negative meson, positive meson, negatron, positron, neutrino and antineutrino respectively.

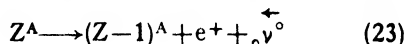
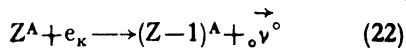
The meson may be described by a scalar function if it be of zero spin and by a vector function if it be of spin one. The scalar and vector meson theories predict distribution of the Fermi type.

### 9. Inversion

The phenomenon of inversion involves two variations of the beta process : either an electron of the extra-nuclear configuration, very usually a K-electron, of the atom may be captured by the nucleus with the emission of a neutrino, or an electron in negative energy state may be absorbed by the nucleus with the vacancy so caused being equivalent to a positron accompanied by antineutrino. In both these variations of the beta process, the nuclear charge is reduced by one unit.

In a nucleus which is unstable with respect to its lower isobar, a nucleon from a proton eigen state changes into a neutron eigen state by emitting a neutrino or an antineutrino

and absorbing an electron (a K-electron) or emitting a positron respectively. These variations of the beta process may be represented thus :



where  $Z$ ,  $A$ ,  $e_K$ ,  $e^+$ ,  ${}_0\nu^0$  and  ${}_0\bar{\nu}^0$  are atomic number, mass number, K-electron, positron, neutrino and antineutrino respectively.

It is of great importance to determine the relative probability of these two processes taking place in this type of radioactive nuclei. In the process given by (22), let the energy released be denoted by  $E$ , the available energy in the process by  $E_0$  and the ionizing energy of a K-electron by  $E_K$ . Then assuming statistical conservation of energy,  $E = E_0 - E_K$ . The process, represented by (23), being the absorption of an electron in negative energy state, is analogous to the process represented by (22).

The probability of the absorption of an electron is proportional to the probability density of the electron in the neighbourhood of the nucleus, i.e. to  $|\phi|^2$ ,  $\phi$  being the wave function of the electron, and to the statistical weight of the final state which is proportional to the square of the energy of neutrino, assuming its rest mass is zero (negligibly small). Hence the capture probability  $P_K$  may be written as

$$P_K = A E_\nu^3 |\phi|^2 \quad (24)$$

where  $A$  is a constant depending upon the kind of transition and  $E_\nu$  the neutrino energy. In the process of K-electron capture, if  $Z$  is small, the wave function of the K-electron may be expressed by

$$\phi_K = \frac{Z^3}{\pi a^3} e^{-\frac{ZR}{a}} \quad (25)$$

where the Bohr radius for K-electron

$$a_K = \frac{h^2}{4\pi^2 m e^2} = \frac{\hbar^2}{m e^2}$$

The completed K-level has two electrons and

$$|\phi|^2 = \frac{Z^3}{\pi a^3}$$

at the centre. Hence the expression (24) for the probability of the K-electron capture may be written in the form :

$$P_K = \frac{2AZ^3 E_\nu^2}{\pi a^3} \quad (26)$$

Now, to determine the probability of positron emission, consider an electron in negative energy state. There are two of them for each cell of volume  $h^3 = 8\pi^3 \hbar^3$  in a phase space. Hence, for positron emission, the probability of finding an electron in negative energy state per unit volume of momentum space is

$$\frac{2}{8\pi^3 \hbar^3} = \frac{1}{4\pi^3 \hbar^3}$$

Therefore, the probability of an electron in negative energy state being absorbed by the nucleus which is equivalent to the probability of the emission of a positron in the momentum range between  $p_x$  and  $p_x + dp_x$ ,  $p_y$  and  $p_y + dp_y$  and  $p_z$  and  $p_z + dp_z$  is given by

$$P_{\beta+} = \frac{AE_v^2}{4\pi^3 \hbar^3} dp_x dp_y dp_z \quad (27)$$

The total probability for positron emission may be obtained by integrating from  $p_x = p_y = p_z = p_0$  to  $E_v = 0$ . Since an energy of  $2mC^2$  is required for an electron in negative kinetic energy state to be removed, the energy of neutrino may be expressed in relativistic units by

$$E_v = E_0 - mC^2 - C\sqrt{p^2 + m^2 C^2},$$

where  $p^2 = p_x^2 + p_y^2 + p_z^2$ . Consider  $\int dp_x dp_y dp_z = 4\pi p^2 dp$  and let  $p_0 = C\sqrt{m^2 C^2 + p^2} = E_0 - mC^2$ . Then the ratio of probabilities of a K-electron capture and positron emission is

$$\frac{P_K}{P_{\beta+}} = 2\pi \left( \frac{2\hbar}{\alpha_K} \right)^3 \frac{E_v^2}{\int_{p_0}^0 E_v^2 p^2 dp} \quad (28)$$

Since  $\frac{\hbar}{\alpha} = \frac{e^2}{\hbar C} mC = \frac{1}{137} mC$  the expression (28) becomes :

$$\frac{P_K}{P_{\beta+}} = 2\pi (Z\alpha)^3 \frac{E_v^2 m^3 C^3}{\int_{p_0}^0 E_v^2 p^2 dp} \quad (29)$$

Thus (28) or (29) represents the Fermi expression for the ratio of probabilities of the K-electron capture and positron emission.

In the Konopinski-Uhlenbeck theory of inversion, the probability of electron capture contains the factor  $|\text{grad } \phi|^2$  instead of the factor  $|\phi|^2$ . With electrons in negative energy state, this factor introduces  $\frac{p^2}{\hbar^2}$  and with K-electrons a factor  $\frac{Z^2}{\alpha^2}$ , so that the

ratio of probabilities of the K-electron capture and positron emission is

$$\frac{P_K}{P_{\beta+}} = 2\pi (Z\alpha)^5 \frac{E_v^2 m^5 C^5}{\int_{p_0}^0 E_v^2 p^4 dp} \quad (30)$$

If the isotopes under consideration be of large  $Z$ , a correction factor arising from the effect of Coulomb field on the wave functions must be introduced in the Fermi expressions (28) and (29) and likewise in the Konopinski-Uhlenbeck expression (30) for the ratio of probabilities of K-electron capture and positron emission.

These theories of inversion for allowed transition, outlined above, give widely differing values for the ratio of probabilities of K-electron capture and positron emission for this type of radioactive nuclei.

In a nucleus which is unstable to its immediately lower isobar, if there is not sufficient energy to cause positron emission, an electron of the K-shell is absorbed by the nucleus and the K-shell will subsequently be filled giving rise to the emission of an X-ray quantum. This quantum is characteristic of the K-radiation of an atom whose number is one below that of the original one, inasmuch as the capture of negative charge reduces the nuclear charge and, therefore, the atomic number by one unit. This characteristic quantum is the distinguishing external effect produced by K-electron capture phenomenon and was the first evidence of this phenomenon.

This phenomenon was observed by Alvarez<sup>15</sup> and subsequently also by others<sup>16</sup>. Alvarez found that  ${}^{48}_{22}\text{Ti}$  bombarded by deuterons emitted a radiation whose absorption coefficient agreed with that of titanium K-characteristic radiation. The active isotope produced by the Ti-d-n reaction was an isotope of vanadium  ${}^{49}_{23}\text{Va}$ , the half life of which was 600 days<sup>17</sup>, while the radiation emitted was titanium K-characteristic X-radiation.

It was found that vanadium<sup>49</sup> was only a K-electron capture disintegrating isotope without positron emission. It was, however, found that there were a few isotopes disintegrating both by K-electron capture and positron emission. An experimental study of this type of active isotopes could prove a touch-stone to test the validity of the two theories of inversion which give very widely diverging values. Hence a measurement of the ratio of probabilities of K-electron capture processes and positron emission processes

of cadmium<sup>107</sup> (<sup>109</sup>?), the half life of which was 6.7 hours, was undertaken by a number of workers<sup>18</sup>. It was found that this isotope, which was disintegrating both by K-electron capture and positron emission, was of an allowed transition probability and that the ratio of K-electron processes to positron emission processes was

$$\frac{P_K}{P_{\beta+} \text{ (observed)}} = 320 \pm 20$$

This observed value, which is in good agreement with the value predicted by the Fermi theory given by

$$\frac{P_K}{P_{\beta+} \text{ (Fermi)}} = 342,$$

is at great variance with the value

$$\frac{P_K}{P_{\beta+} \text{ (K.U.)}} = 19800$$

given by the Konopinski-Uhlenbeck theory, which, therefore, is ruled out.

The author wishes to record his appreciation of the active co-operation of his scientific assistants, U. C. Gupta, A. Sagar, P. N. Sundaram, R. Sundaram and S. K. Suri, who helped him in compiling and calculating the data given in Appendix I,

and plotting a number of Sargent diagrams, each covering a wide range of beta-decaying isotopes.

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## APPENDIX I

### BETA-DECAYING ISOTOPES

( See Section 7, Page 403 )

ISOTOPE	SYMBOL		HALF-LIFE ( Sec. )		DECAY CONST. ( Sec. <sup>-1</sup> )	ENERGY MEV.	RADIATION
Hydrogen	H	1	3	10 <sup>9</sup>	6.9 × 10 <sup>-10</sup>	0.015	β <sup>-</sup>
Helium	He	2	6	0.8	8.622 × 10 <sup>-1</sup>	3.7	
Lithium	Li	3	8	0.88	7.840 × 10 <sup>-1</sup>	12.0	
Beryllium	Be(1)	4	7	5 × 10 <sup>6</sup>	1.380 × 10 <sup>-7</sup>		K
	Be(2)	"	7	5 × 10 <sup>7</sup>	1.380 × 10 <sup>-8</sup>		K, γ
	Be	"	10	10 <sup>13</sup>	6.902 × 10 <sup>-14</sup>	0.5	β <sup>-</sup> , γ
Boron	B	5	12	0.022	31.36	12.0	β <sup>-</sup>
Carbon	C	6	10	8.8	7.84 × 10 <sup>-2</sup>	3.4	β <sup>+</sup>
	C	"	11	1230	5.611 × 10 <sup>-5</sup>	0.95	β <sup>+</sup>
	C	"	14	> 10 <sup>13</sup>	~ 6.9 × 10 <sup>-13</sup>	0.145	β <sup>-</sup>
Nitrogen	N	7	13	3000(?)	2.3 × 10 <sup>-4</sup>	...	β <sup>+</sup> , γ
	N	"	16	8	8.628 × 10 <sup>-2</sup>	6.0(?)	β <sup>-</sup>
Oxygen	O	8	15	126	5.321 × 10 <sup>-3</sup>		β <sup>+</sup>
	O		19	31	2.22 × 10 <sup>-2</sup>		

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

ISOTOPE	SYMBOL	Z	A	HALF-LIFE (Sec.)	DECAY CONST. (Sec. <sup>-1</sup> )	ENERGY MEV.	RADIATION
Fluorine	F	9	17	70	$9.86 \times 10^{-3}$	2.1	$\beta^+$
	F	"	18	6720	$1.027 \times 10^{-4}$	0.7	$\beta^+$
	F	"	20	12	$5.749 \times 10^{-2}$	5.0	$\beta^-, \gamma$
Neon	Ne	10	19	20.3	$3.399 \times 10^{-2}$	2.20	$\beta^+$
	Ne	"	23	40	$1.723 \times 10^{-2}$	4.1	$\beta^-$
Sodium	Na	11	21	23	$3.000 \times 10^{-2}$	...	...
	Na	"	22	$> 10^8$	$6.897 \times 10^{-9}$	0.58	$\beta^+, \gamma$
	Na	"	24	$5.3 \times 10^4$	$1.302 \times 10^{-5}$	1.4	$\beta^-, \gamma$
	Na	"	25	62	$1.11 \times 10^{-2}$	2.8	$\beta^-, \gamma$
Magnesium	Mg	12	23	11.6	$5.950 \times 10^{-2}$	2.82	$\beta^+$
	Mg	"	27	612	$1.127 \times 10^{-3}$	1.8	$\beta^-, \gamma$
Aluminium	Al	13	26	7.0	$9.860 \times 10^{-2}$	2.99	$\beta^+$
	Al	"	28	144	$4.791 \times 10^{-3}$	3.3	$\beta^-, \gamma$
	Al	"	29	402	$1.716 \times 10^{-3}$	2.5	$\beta^-$
Silicon	Si	14	27	4.92	$1.409 \times 10^{-1}$	3.74	$\beta^+$
	Si	"	31	10200	$6.9 \times 10^{-4}$	1.8	$\beta^-$
Phosphorus	P	15	29	4.6	$1.500 \times 10^{-1}$	3.63	$\beta^+$
	P	"	30	153	$4.50 \times 10^{-3}$	3.0	$\beta^+$
	P	"	32	$1.2 \times 10^6$	$5.751 \times 10^{-7}$	1.69	$\beta^-$
Sulphur	S	16	31	3.18	$2.156 \times 10^{-1}$	3.90	$\beta^+$
	S	"	35	$7.52 \times 10^6$	$9.075 \times 10^{-8}$	0.107	$\beta^-$
Chlorine	Cl	17	33	2.8	$2.46 \times 10^{-1}$	...	$\beta^+$
	Cl	"	34	1980	$3.484 \times 10^{-4}$	2.5	$\beta^+$
	Cl	"	36	$> 10^8$	$6.902 \times 10^{-9}$	0.66	$\beta^+, K, \beta^-$
	Cl	"	38	2220	$3.136 \times 10^{-4}$	1.1, 5.0	$\beta^-, \gamma$
Argon	A	18	35	1.91	$3.63 \times 10^{-1}$	4.38	$\beta^+$
	A	"	39	240	$2.874 \times 10^{-3}$	...	$\beta^-$
	A	"	41	6600	$1.046 \times 10^{-4}$	1.5	$\beta^-, \gamma$
Potassium	K	19	38	462	$1.494 \times 10^{-3}$	2.3	$\beta^+$
	K	"	40	$4.5 \times 10^{16}$	$1.534 \times 10^{-17}$	0.40; 0.70	$\beta^-$
	K	"	42	$4.5 \times 10^4$	$1.534 \times 10^{-5}$	3.5	$\beta^-$
	K	"	43, 44	1080	$6.388 \times 10^{-4}$	...	$\beta^-$
Calcium	Ca	20	39	270	$2.555 \times 10^{-3}$	...	$\beta^+$
	Ca	"	41	$7.34 \times 10^5$	$9.4 \times 10^{-6}$	...	K, $\gamma, e^-$
	Ca	"	45	$1.55 \times 10^7$	$4.45 \times 10^{-8}$	0.2	$\beta^-, \gamma$
	Ca	"	49	$9 \times 10^3$	$7.66 \times 10^{-5}$	2.3	$\beta^-, \gamma$
	Ca	"	49	1800	$3.83 \times 10^{-4}$	...	$\beta^-$
Scandium	Sc	21	41	0.87	$7.93 \times 10^{-1}$	4.94	$\beta^+$
	Sc	"	42	$1.17 \times 10^6$	$5.897 \times 10^{-7}$	1.4	$\beta^+$
	Sc	"	43	$1.44 \times 10^4$	$4.791 \times 10^{-5}$	0.4; 1.4	$\beta^+, \gamma$
	Sc	"	44	$1.87 \times 10^5$	$3.689 \times 10^{-6}$	...	$e^-, \gamma$
	Sc	"	44	$1.48 \times 10^4$	$4.67 \times 10^{-5}$	1.45	$\beta^+, \gamma$
	Sc	"	46	$7.34 \times 10^6$	$9.4 \times 10^{-7}$	0.26; 1.5	$\beta^-, \gamma, K$
	Sc	"	47	$2.27 \times 10^5$	$3.039 \times 10^{-6}$	1.1	$\beta^-, \gamma$
	Sc	"	48	$1.58 \times 10^5$	$4.34 \times 10^{-6}$	0.64	$\beta^-, \gamma$
	Sc	"	49	$3.42 \times 10^3$	$1.480 \times 10^{-4}$	1.8	$\beta^-$
	Sc	"	?	$2.94 \times 10^5$	$2.346 \times 10^{-6}$	0.46	$\beta^-$
	Sc	"	?	?	?	?	?
Titanium	Ti	22	45	$1.1 \times 10^4$	$6.275 \times 10^{-5}$	1.2	$\beta^+$
	Ti	"	51	174	$3.965 \times 10^{-3}$	...	$\beta^-, \gamma$
	Ti	"	51	$6.2 \times 10^6$	$1.113 \times 10^{-7}$	0.36	$\beta^+$
Vanadium	V	23	47	1980	$3.487 \times 10^{-4}$	1.9	$\beta^+$
	V	"	48	$> 1.4 \times 10^6$	$< 4.928 \times 10^{-7}$	1.0	$\beta^+, K, \gamma$
	V	"	49	$5.18 \times 10^7$	$1.33 \times 10^{-8}$	...	K
	V	"	50	$1.33 \times 10^4$	$5.187 \times 10^{-5}$	...	$\beta^+$
	V	"	52	234	$2.968 \times 10^{-3}$	2.05	$\beta^-$
Chromium	Cr	24	49	2514	$2.748 \times 10^{-4}$	1.45	$\beta^+, \gamma$
	Cr	"	51	$2.29 \times 10^6$	$3.01 \times 10^{-7}$	...	K, $\gamma, e^-$
Manganese	Mn	25	51	2760	$2.50 \times 10^{-4}$	2.0	$\beta^+$
	Mn	"	52	1260	$5.321 \times 10^{-4}$	2.2	$\beta^+, \gamma$

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

ISOTOPE	SYMBOL	Z	A	HALF-LIFE (Sec.)	DECAY CONST. (Sec. <sup>-1</sup> )	ENERGY MEV.	RADIATION
Manganese	Mn	25	52	$5.62 \times 10^5$	$1.23 \times 10^{-6}$	0.77	$\beta^+$ , $\gamma$
	Mn	"	54	$2.68 \times 10^7$	$2.574 \times 10^{-8}$	...	K, $\gamma$
	Mn	"	56	$9.32 \times 10^3$	$7.367 \times 10^{-4}$	0.75 ; 2.86 ; 1.09	$\beta^-$ , $\gamma$
Iron	Fe	26	53	534	$1.3108 \times 10^{-3}$	...	$\beta^+$
	Fe	"	55	$\sim 1.26 \times 10^8$	$5.321 \times 10^{-9}$	...	K, $e^-$
	Fe	"	59	$3.8 \times 10^6$	$1.81 \times 10^{-7}$	0.26 ; 0.46	$\beta^-$ , $\gamma$
Cobalt	Co	27	55	$6.55 \times 10^6$	$3.797 \times 10^{-4}$	1.50	$\beta^+$ , $\gamma$
	Co	"	56	$6.22 \times 10^6$	$1.109 \times 10^{-7}$	1.2	$\beta^+$ , $\gamma$ , K
	Co	"	57	$2.33 \times 10^7$	$2.98 \times 10^{-8}$	2.6	K, $\gamma$ , $e^-$ , $\beta^+$
	Co	"	58	$6.221 \times 10^3$	$1.109 \times 10^{-4}$	0.470	$\beta^+$ , $\gamma$
	Co	"	60	$1.67 \times 10^8$	$4.13 \times 10^{-9}$	0.31	$\gamma$
	Co	"	60	640	$1.078 \times 10^{-3}$	1.3	$\beta^-$ , $\gamma$
Nickel	Ni	28	57	$1.3 \times 10^5$	$5.342 \times 10^{-6}$	0.67	$\beta^+$
	Ni	"	59	$4.72 \times 10^8$	$1.48 \times 10^{-9}$	0.05	$\beta^+$
	Ni	"	63	9350	$7.379 \times 10^{-5}$	1.9	$\beta^-$ , $\gamma$
Copper	Cu	29	58, 60	81	$8.51 \times 10^{-3}$	...	$\beta^+$
	Cu	"	61	$1.3 \times 10^4$	$5.342 \times 10^{-5}$	0.9	$\beta^+$
	Cu	"	61	$1.3 \times 10^4$	$5.342 \times 10^{-5}$	...	K
	Cu	"	62	63	$1.064 \times 10^{-3}$	2.6	$\beta^+$
	Cu	"	64	$4.6 \times 10^4$	$1.501 \times 10^{-5}$	0.58( $\beta^-$ ); 0.66( $\beta^+$ )	$\beta^+$ or $\beta^-$ or K
	Cu	"	66	300	$2.301 \times 10^{-3}$	2.9	$\beta^-$
Zinc	Zn	30	63	2280	$3.0026 \times 10^{-4}$	2.32	$\beta^+$
	Zn	"	65	$2.16 \times 10^7$	$3.29 \times 10^{-8}$	0.4( $\beta^+$ )	$\beta^+$ , K, $\gamma$ , $e^-$
	Zn	"	69	3420	$2.018 \times 10^{-4}$	1.0	$\beta^-$
Gallium	Ga	31	64	2880	$2.39 \times 10^{-4}$	...	$\beta^+$
	Ga	"	65	900	$7.66 \times 10^{-3}$	...	K, $e^-$
	Ga	"	67	$2.99 \times 10^5$	$2.31 \times 10^{-6}$	...	K, $\gamma$ , $e^-$
	Ga	"	66	$3.4 \times 10^4$	$2.29 \times 10^{-5}$	3.1	$\beta^+$
	Ga	"	68	4080	$1.691 \times 10^{-4}$	1.9	$\beta^+$
	Ga	"	70	1140	$6.054 \times 10^{-4}$	1.70	$\beta^-$ , $\gamma$
	Ga	"	72	$5 \times 10^4$	$1.38 \times 10^{-5}$	2.6	$\beta^-$ , $\gamma$
Germanium	Ge	32	71	$1.1 \times 10^5$	$6.275 \times 10^{-6}$	1.2	$\beta^+$
	Ge	"	75	4860	$1.428 \times 10^{-4}$	1.1	$\beta^-$
	Ge	"	77	$2.9 \times 10^4$	$2.380 \times 10^{-5}$	1.9	$\beta^-$
Arsenic	As	33	73	$1.8 \times 10^5$	$3.835 \times 10^{-6}$	0.6	$\beta^+$
	As	"	74	$1.4 \times 10^6$	$4.928 \times 10^{-7}$	1.3( $\beta^-$ ) ; 0.9( $\beta^+$ )	$\beta^-$ , $\beta^+$ , $\gamma$
	As	"	78	3780	$1.825 \times 10^{-4}$	1.4	$\beta^-$ , $\gamma$
Selenium	Se	34	81	1140	$6.054 \times 10^{-4}$	1.5	$\beta^-$
Bromium	Br	35	78	384	$1.796 \times 10^{-3}$	2.3	$\beta^+$ , $\gamma$
	Br	"	80	1080	$6.398 \times 10^{-4}$	2.0	$\beta^-$ , ?
	Br	"	82	$1.2 \times 10^5$	$5.747 \times 10^{-6}$	0.465	$\beta^-$ , $\gamma$
	Br	"	83	8400	$8.214 \times 10^{-4}$	1.05	$\beta^-$
Krypton	Kr	36	79	$1.2 \times 10^5$	$5.751 \times 10^{-6}$	...	$\beta^+$
	Kr	"	...	...	...	...	...
	Kr	"	88	$1.1 \times 10^4$	$6.275 \times 10^{-5}$	2.5	$\beta^-$
Rubidium	Rb	37	86	$1.7 \times 10^6$	$4.588 \times 10^{-7}$	1.56	$\beta^-$
	Rb	"	87	$6 \times 10^{18}$	$1.150 \times 10^{-19}$	0.132 or 0.25	$\beta^-$
	Rb	"	88	$1.1 \times 10^4$	$6.275 \times 10^{-5}$	5.1	$\beta^-$
	Rb	"	89	900	$7.666 \times 10^{-4}$	3.8	$\beta^-$ , $\gamma$
Strontium	Sr	38	89	$4.8 \times 10^6$	$1.437 \times 10^{-7}$	1.50 or 1.32	$\beta^-$
Yttrium	Y	39	87	$5.04 \times 10^4$	$1.37 \times 10^{-5}$	0.5	$e^-$ , $\gamma$
	Y	"	88	7200	$9.581 \times 10^{-5}$	1.2	$\beta^+$
	Y	"	90	$2.2 \times 10^5$	$3.137 \times 10^{-6}$	2.6	$\beta^-$
	Y	"	91	$4.92 \times 10^6$	$1.406 \times 10^{-7}$	1.6	$\beta^-$ , $\gamma$
Zirconium	Zr	40	89	$2.8 \times 10^5$	$2.465 \times 10^{-6}$	1.0	$\beta^+$
	Zr	"	89	27	$2.55 \times 10^{-2}$	...	$e^-$ , $\gamma$
	Zr	"	93	$5.44 \times 10^6$	$1.256 \times 10^{-7}$	0.25 or 0.57 or 0.29	$\beta^-$ , $\gamma$
	Zr	"	95	$6.12 \times 10^4$	$1.126 \times 10^{-5}$	1.0	$\beta^-$
	Zr	"	97	$3.6 \times 10^2$	$1.917 \times 10^{-3}$	1.9	$\beta^-$
	Zr	"	97	$5.4 \times 10^3$	$1.277 \times 10^{-4}$	1.5	$\beta^-$
	Zr	"	97	$2.52 \times 10^5$	$2.62 \times 10^{-6}$	1.17	$\beta^-$

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

ISOTOPE	SYMBOL	Z	A	HALF-LIFE (Sec.)	DECAY CONST. (Sec. <sup>-1</sup> )	ENERGY MEV.	RADIATION
Columbium	Cb	41	92	$9.5 \times 10^5$	$7.28 \times 10^{-8}$	1.38 or 0.59	$\beta^-$ , $\gamma$
	Cb	"	94	396	$1.743 \times 10^{-4}$	1.4	$\beta^-$ , $\gamma$
	Cb	"	95	4500	$1.533 \times 10^{-4}$	1.0	$\beta^-$
Molybdenum	Mo	42	91	1020	$6.764 \times 10^{-4}$	...	$\beta^+$
	Mo	"	99	$2.4 \times 10^5$	$2.874 \times 10^{-6}$	1.5	$\beta^-$ , $\gamma$
	Mo	"	101	876	$7.88 \times 10^{-3}$	1.8, 1.0, 2.2	$\beta^-$ , $\gamma$
Masurium	Ma	43	101	$8.4 \times 10^2$	$8.21 \times 10^{-4}$	1.1, 1.3	$\beta^-$ , $\gamma$
	Ma	"	101	$3.30 \times 10^3$	$2.089 \times 10^{-4}$	2.5	$\beta^-$ , $\gamma$
	Ma	"	96	$9.72 \times 10^3$	$7.18 \times 10^{-4}$	...	$\beta^+$ (?)
	Ma	"	101?	18	$3.83 \times 10^2$	...	$\beta^-$
	Ma	"	101?	$1.31 \times 10^5$	$5.34 \times 10^{-6}$	...	$\beta^-$
	Ma	"	99	$2.38 \times 10^4$	$2.93 \times 10^{-5}$	...	$e^-$ , $\gamma$
Ruthenium	Ru	44	105	$1.44 \times 10^4$	$4.6 \times 10^{-5}$	1.5	$\beta^-$
	Ru	"	105	240	$2.874 \times 10^{-3}$	4.0	$\beta^-$
Rhodium	Rh	45	102	$1.81 \times 10^7$	$3.828 \times 10^{-8}$	1.1 ( $\beta^-$ )	$\beta^-$ , $\beta^+$ , $\gamma$
	Rh	"	104	44	$1.568 \times 10^{-3}$	2.3	$\beta^-$
	Rh	"	105	$1.22 \times 10^5$	$5.70 \times 10^{-6}$	0.5	$\beta^-$
Palladium	Pd	46	107	$4.7 \times 10^4$	$1.468 \times 10^{-5}$	1.03	$\beta^-$
	Pd	"	111	1560	$4.424 \times 10^{-4}$	3.5	$\beta^-$
Silver	Ag	47	106	1470	$4.696 \times 10^{-4}$	2.04	$\beta^+$ (?)
	Ag	"	108	138	$5.001 \times 10^{-3}$	2.8	$\beta^-$
	Ag	"	110	22	$3.136 \times 10^{-3}$	2.8	$\beta^-$ , $\gamma$
	Ag	"	112	$1.2 \times 10^4$	$5.751 \times 10^{-5}$	2.2	$\beta^-$ , $\gamma$
	Ag	"	111	$6.48 \times 10^5$	$1.07 \times 10^{-6}$	0.8	$\beta^-$
Cadmium	Cd	48	115	$2.2 \times 10^5$	$3.136 \times 10^{-6}$	1.11	$\beta^-$ , $\gamma$
	Cd	"	115(?)	$3.46 \times 10^6$	$2.006 \times 10^{-7}$	0.095	$\beta^-$ , $\gamma$
	Cd	"	117	$1.35 \times 10^4$	$5.1 / \times 10^{-5}$	...	$\beta^-$
Indium	In	49	110	3900	$1.781 \times 10^{-4}$	1.6	$\beta^+$
	In	"	111	1380	$5.001 \times 10^{-4}$	...	$\beta^+$ , $\gamma$
	In	"	114	72	$9.580 \times 10^{-3}$	1.98	$\beta^-$
	In	"	116	13 or 3240	$5.342 \times 10^{-2}$ or $2.12 \times 10^{-4}$	2.8 or .85	$\beta^-$ or $\beta^-$ , $\gamma$
	In	"	117	7020	$9.844 \times 10^{-5}$	1.73	$\beta^-$ , $\gamma$ , $e^-$
Tin	Sn	50	125	540	$1.28 \times 10^{-3}$	...	$\beta^-$
Antimony	Sb	51	120	1020	$6.764 \times 10^{-4}$	1.53	$\beta^+$
	Sb	"	122	$2.4 \times 10^5$	$2.884 \times 10^{-6}$	0.81	$\beta^-$ , $\gamma$
	Sb	"	122	$> 2.4 \times 10^5$	$\sim 2.884 \times 10^{-6}$	1.64	$\beta^-$ , $\gamma$
Tellurium	Te	52	129	4320	$1.6 \times 10^{-4}$	...	$\beta^-$
	Te	"	131	1500	$4.60 \times 10^{-4}$	...	$\beta^-$
Iodine	I	53	126	$1.1 \times 10^6$	$6.272 \times 10^{-7}$	1.1	$\beta^-$ , $\gamma$
	I	"	128	1499.40	$4.61 \times 10^{-4}$	1.85, 1.05, 2.10	$\beta^-$ , $\gamma$
	I	"	130	$4.54 \times 10^4$	$1.516 \times 10^{-5}$	0.61, 1.03	$\beta^-$ , $\gamma$
	I	"	131	$6.9 \times 10^5$	$1.00 \times 10^{-6}$	...	$\beta^-$ , $\gamma$
	I	"	133	$7.92 \times 10^4$	$8.78 \times 10^{-5}$	1.1	$\beta^-$
Xenon	Xe	54	135	936	$7.371 \times 10^{-3}$	0.7 or 0.6	$\beta^-$ , $\gamma$
	Xe	"	137	204	$3.39 \times 10^{-3}$	4.0	$\beta^-$
	Xe	"	135	$3.38 \times 10^4$	$2.403 \times 10^{-5}$	0.95, 0.90	$\beta^-$ , $\gamma$
Cesium	Cs	55	134	$5.35 \times 10^7$	$1.29 \times 10^{-8}$	0.9	$\beta^-$ , $\gamma$
	Cs	"	134	$1.1 \times 10^4$	$6.275 \times 10^{-5}$	1.0	$\beta^-$
	Cs	"	138	1980	$3.48 \times 10^{-4}$	2.6	$\beta^-$
Barium	Ba	56	139	5160	$1.336 \times 10^{-4}$	1 or 2.3	$\beta^-$ , $\gamma$
	Ba	"	140	$1.08 \times 10^5$	$6.39 \times 10^{-5}$	1.2	$\beta^-$
Lanthanum	La	57	140	$1.44 \times 10^5$	$4.80 \times 10^{-6}$	1.41	$\beta^-$ , $\gamma$
Cerium	Ce	58	139	$1.26 \times 10^2$	$5.322 \times 10^{-3}$	...	$\beta^+$
	Ce	"	143	$1.3 \times 10^5$	$5.342 \times 10^{-6}$	...	$\beta^-$ (?)
	Ce	"	141	$2.59 \times 10^6$	$2.67 \times 10^{-7}$	0.65	$\beta^-$ , $\gamma$
Praseodymium	Pr	59	140	210	$3.28 \times 10^{-3}$	2.4	$\beta^+$
	Pr	"	142	$6.95 \times 10^4$	$1.010 \times 10^{-5}$	2.14	$\beta^-$ , $\gamma$
	Pr	"	143	$1.17 \times 10^6$	$6.115 \times 10^{-7}$	0.95	$\beta^-$
	Pr	"	143(?)	1020	$6.9 \times 10^{-4}$	3.1	$\beta^-$

APPENDIX I — BETA-DECAYING ISOTOPES — *continued*

ISOTOPE	SYMBOL	Z	A	HALF-LIFE (Sec.)	DECAY CONST. (Sec. <sup>-1</sup> )	ENERGY MEV.	RADIATION
Neodymium	Nd	60	141	$9 \times 10^3$	$7.66 \times 10^{-5}$	0.78	$\beta^+$
Illinium	Il	61	?	$4.58 \times 10^5$	$1.506 \times 10^{-6}$	2	$\beta^-, \gamma$
	Il	..	?	$9.72 \times 10^3$	$7.098 \times 10^{-6}$	2	$\beta^-, \gamma$
	Il	..	?	$1.38 \times 10^6$	$5.000 \times 10^{-7}$	1.7	$\beta^-, \gamma$
Samarium	Sm	62	154?	1260	$5.321 \times 10^{-4}$	1.8	$\beta^-$
Europium	Eu	63	150	$9.72 \times 10^4$	$7.098 \times 10^{-5}$	...	$\beta^+$
	Eu	..	154	$1.57 \times 10^2$ to $2.52 \times 10^8$	$4.40 \times 10^{-9}$ or $2.62 \times 10^{-9}$	0.9	$\beta^-, \gamma$
	Eu	..	152	$3.31 \times 10^4$	$2.091 \times 10^{-5}$	1.88( $\beta^-$ )	$\beta^-, \gamma, e^-$
Terbium	Tb	65	160	$6.22 \times 10^6$	$1.193 \times 10^{-7}$	0.70	$\beta^-, \gamma$
Dysprosium	Dy	66	165	9000	$7.666 \times 10^{-5}$	1.20	-
Holmium	Ho	67	166	$1.3 \times 10^5$	$5.342 \times 10^{-6}$	1.6	$\beta^-$
Lutecium	Lu	71	176, 177	$1.22 \times 10^4$	$5.855 \times 10^{-5}$	1.15	$\beta^-$
	Lu	..	176, 177	$5.70 \times 10^5$	$1.22 \times 10^{-6}$	0.44	$\beta^-$
	Lu	..	176	$2.3 \times 10^{18}$	$3 \times 10^{-19}$	0.215, 0.40	$\beta^-$
Tantalum	Ta	73	182	$8.38 \times 10^6$	$8.233 \times 10^{-7}$	1.0, 0.98, 0.32 0.050	$\beta^-, \gamma$
Tungsten	W	74	185	$6.7 \times 10^6$	$1.030 \times 10^{-7}$	0.55—0.65 0.64—0.72	$\beta^-, \gamma$
	W	..	187	$8.3 \times 10^4$	$8.915 \times 10^{-6}$	1.4	$\beta^-, \gamma$
Rhenium	Re	75	186	$3.2 \times 10^5$	$2.157 \times 10^{-6}$	1.05	$\beta^-$
	Re	..	188	$6.5 \times 10^4$	$1.062 \times 10^{-5}$	2.5	$\beta^-$
Osmium	Os	76	191	$1.2 \times 10^5$	$5.748 \times 10^{-6}$	1.5	$\beta^-, \gamma$
	Os	..	193	$3.9 \times 10^6$	$1.770 \times 10^{-7}$	0.35	$\beta^-, \gamma$
Iridium	Ir	77	194	$6.8 \times 10^4$	$1.014 \times 10^{-5}$	2.2	$\beta^-, \gamma$
	Ir	..	192, 194	$518 \times 10^6$	$1.33 \times 10^{-7}$	..	$\beta^-, \gamma$
Platinum	Pt	78	197	$6.48 \times 10^4$	$1.064 \times 10^{-5}$	0.65, 0.72	$\beta^-$
	Pt	..	199	1860	$3.7 \times 10^{-4}$	1.8	$\beta^-$
Gold	Au	79	196	$4 \times 10^5$	$1.726 \times 10^{-6}$	0.36	$\beta^-, \gamma$
	Au	..	198	$2.3 \times 10^5$	$3 \times 10^{-6}$	0.8	$\beta^-, \gamma$
	Au	..	199	$2.85 \times 10^5$	$2.429 \times 10^{-6}$	1.01	$\beta^-, \gamma$
Mercury	Hg	80	203, 205	$4.45 \times 10^6$	$1.550 \times 10^{-7}$	0.46	$\beta^-, \gamma$
	Hg	..	205	330	$2.9 \times 10^{-3}$	1.62	$\beta^-$
Thallium	Tl	81	204	253	$2.727 \times 10^{-3}$	1.6	$\beta^-$
	Tl	..	206	$1.1 \times 10^8$	$6.27 \times 10^{-9}$	0.87	$\beta^-$
	AcC'	..	207	291	$2.371 \times 10^{-3}$	1.47	$\beta^-, \gamma$
	ThC''	..	208	186	$5.480 \times 10^{-3}$	1.82	$\beta^-, \gamma$
	RaC''	..	210	79.2	$1.209 \times 10^{-3}$	1.80	$\beta^-$
Lead	Pb	82	203	615.00	$1.122 \times 10^{-3}$	1.66	$\beta^+$
	Pb	..	209	$10.8 \times 10^3$	$6.389 \times 10^{-4}$	0.7 ; 0.75	$\beta^-$
	RaD	..	210	$6.93 \times 10^8$	$1.00 \times 10^{-9}$	0.255	$\beta^-, \gamma$
	AcB	..	211	2100	$3.287 \times 10^{-4}$	0.5 ; 1.4	$\beta^-, \gamma$
	ThB	..	212	$3.8 \times 10^4$	$1.817 \times 10^{-5}$	0.36	$\beta^-, \gamma$
	RaB	..	214	7608	$9.069 \times 10^{-4}$	0.65	$\beta^-, \gamma$
	RaE	83	210	$4.3 \times 10^5$	$1.604 \times 10^{-6}$	...	$\beta^-$
	87 (Ack)	87	223	1260	$5.33 \times 10^{-4}$	...	$\beta^-, \gamma$
	Ms Th	88	228	$2.1 \times 10^8$	$3.193 \times 10^{-9}$	0.053	$\beta^-$
	Ms Th <sub>2</sub>	89	228	$2.2 \times 10^4$	$3.137 \times 10^{-5}$	1.55, 4.5	$\beta^-, \gamma$
	Uy	90	231	$8.86 \times 10^4$ or $8.64 \times 10^4$	$7.985 \times 10^{-6}$ or $7.787 \times 10^{-6}$	0.2	$\beta^-$
	Thorium	Th	90	1380	$5.000 \times 10^{-4}$	...	$\beta^-$
	Ux <sub>1</sub>	..	234	$8.68 \times 10^4$ or $8.82 \times 10^4$	$7.823 \times 10^{-6}$ or $7.77 \times 10^{-6}$	0.130, 0.100 0.11, 0.13, 0.20	$\beta^-, \gamma$
Protactinium	Pa	91	233	$2.37 \times 10^6$	$2.911 \times 10^{-7}$	0.4 or 0.23	$\beta^-, \gamma, e^-$
	Uz	..	234	$2.41 \times 10^4$	$2.862 \times 10^{-5}$	0.56, 1.55	$\beta^-, \gamma$
	Ux <sub>2</sub>	..	234	68	$1.014 \times 10^{-2}$	2.32	$\beta^-, \gamma$
Uranium	U	92	237	$6 \times 10^5$	$1.150 \times 10^{-6}$	0.26	$\beta^-, \gamma$
	U	..	239	1410	$4.894 \times 10^{-4}$	0.56, 1.2	$\beta^-, \gamma$
Neptunium	Np	93	235	$> 3.15 \times 10^7$	$< 2.188 \times 10^{-8}$	...	K, $\gamma$
	Np	..	238	$1.728 \times 10^5$	$3.993 \times 10^{-6}$	1.35	$\beta^-, \gamma$
	Np	..	239	...	$3.427 \times 10^{-6}$	0.14 ; 0.78	$\beta^-, \gamma$



# REVIEWS

**History of Dyes and Dyeing in the Bombay Presidency**, by Mr. B. N. Phadke (Dastane Bros.; Home Service Ltd., 456 Raviwar Peth, Poona 2), 1947, pp. 152. Price Rs. 12.

INDIA IS NOW ONE OF THE LEADING COTTON textile manufacturing countries of the world and Bombay province is the home of the cotton textile industry in India. A survey of the dyeing industry in that province is tantamount to giving a correct picture of the industry in the whole of India. Mr. Phadke's book under review is an important contribution to the history of dyes and dyeing — particularly when there is in evidence a keen desire for the establishment of a dyestuff industry in India.

The book is divided into 5 chapters and an appendix containing 11 tables is included at the end.

Chapter I is a historical introduction. The second chapter deals with indigenous dyestuffs. The process of dyeing of cotton, silk, wool, ivory, wood and leather adopted in various parts of the Bombay Presidency are discussed in detail and recent work on vegetable dyes and efforts to revive the indigenous dyestuff industry are also given.

Various methods of application of mineral pigments on textile fibre for producing shades of khaki are fully treated in the third chapter.

The fourth chapter is the most important in that it is concerned with synthetic dyestuffs and methods of application to different types of fibres like cotton, silk, rayon and acetate silk. This chapter is full of useful information regarding the trade names under which the various classes of dyes are sold in the market by different manufacturers, and their extent of popularity in the trade. The classification of the dyes and their application for miscellaneous purposes, for example, (a) yarn printing; (b) writing inks and stamping paste; (c) food colours; (d) lakes and pigments; and (e) paper, wood and leather colouring are very useful.

That the indigenous dyes, with very few exceptions, cannot compete with coal-tar

colours has been well brought out in the last chapter. The circumstances which have led to the decline of the vegetable dyestuff industry, and the reasons therefor, as well as the possibility of revival of a few indigenous dyes like natural indigo and cutch have also been well brought out.

The appendix contains interesting information on the chemical aspects of some of the dyes, the communities engaged in the industry, the variety of textile goods manufactured in various parts of the Presidency and different classes of dyestuffs used therein, list of manufacturers, importers or sellers and trade names, statistics of export-import data and other relevant details.

The get-up of the book is satisfactory. Some more care might have been exercised about the botanical names of the plants listed in table No. 1 of the appendix where there are innumerable mistakes in the names of the plants: *Butea frondosa*, Rox; *Adanantthera payonina*, W; *Hyctanthes arbartristis*; *Sympocos racemosa*, Rox; *Acacia catechu* Will; *Pernelia chamchadalis*, etc. It is understandable why the author should have used the out-of-date name of *Calysaccion longifolium* when *Ochrocarpos longifolius*, Benth & Hook, is found in Cooks *Flora of the Bombay Presidency* which has been listed in the bibliography. It would have been better if the author had referred to up-to-date books on chemistry of natural colouring matters before giving the structural formulae of some of the natural dyes. For example, Brazilin has been given the formula ascribed to it by Pfeiffer whereas it has been admitted that Perkins formula is preferable. Similarly, for catechin Freudenberg's formula is to be preferred to Nierenstein formula given by the author. It is difficult to understand how the accepted Robertson and Brockmann formulae have been discarded by the author for rottlerin. The colouring matter of Henna has been identified to be 2-hydroxy 1-4 naphthaquinone whereas the author has given it as  $C_{10}H_6O_3$ .

The book contains valuable information and supplies a long-felt want.

A. CHARI

**Industrial Profits in India**, by M. C. Munshi (Research Department, Federation of Indian Chambers of Commerce & Industry), 1949, pp. iii+326. Price Rs. 15.

A SCIENTIFIC STUDY OF INDUSTRIAL PROFITS is a valuable aid to the framing of business policy. It gives better insight into business activity in general, apart from indicating trends of corporate earnings. Further, a proper analysis of profit trends is indispensable for deciding questions such as a fair return on capital or the influence of taxation on the production incentive. The author of the publication under review has attempted an analysis of the industrial profits in India during the years 1936-44 and has based his study on a painstaking collection of statistical data. By a close study of the balance-sheets and profit and loss statements of a large number of joint-stock companies, the author has arrived at many interesting findings relating to the progress of some of the major industries and their comparative success or otherwise. For instance, the author shows the high profits in cotton, paper and jute industries followed by sugar and iron and steel, and contrasts how the coal industry, because of transport difficulties and internal weaknesses, could benefit little out of the war except towards the end of the period. "An important feature of our industrial development during the War", says the author, "— in marked contrast with those of some other belligerent countries — was that our industries could earn high profits even when the quantum of their production remained either stationary or showed a marked decline." Again, "Another unmistakable trend that stands out in bold relief from our tables is that all these industries during the War have collected from the pockets of the consumer what they filled the coffers of the Government with."

The enquiry covers the seven major industries and relates to 71 cotton mill companies, 60 jute mill companies, 54 coal mining companies, 48 sugar mill companies, 8 paper mill companies and 2 each in the case of iron and steel and cement industries. The book abounds in numerous tables and graphs revealing trends of net profits, dividends, provision for taxation, contributions to reserves, the ratio of net profits to sales and numerous other facts and figures pertaining to the enquiry. The work will, no doubt, be regarded as a significant contri-

bution to the field of statistics relating to industrial profits in India. It is hoped that the study which ends with 1944 will be brought up to date.

R. B. P.

**Schimmel 1946 Report on Essential Oils, Aromatic Chemicals and Related Materials** (published by Schimmel & Co., Inc., New York), pp. 137. Price \$3.25.

THE 1946 ANNUAL SCHIMMEL REPORT ON *Essential Oils, Aromatic Chemicals and Related Materials* has just become available and appears to be a volume of more than usual interest.

An interesting section is devoted to the pharmacology of essential oils, and contains discussions of their skin-irritating properties and of the anti-histaminic action of citral. The biogenesis of perfume materials comprises another interesting section.

Among the developments in the field of essential oils and perfume chemicals during the year, some of the following may prove of particular interest to readers:

The essential oil distilled from wild cloves collected in the Moluccas was discovered to be quite different in composition from commercial clove oil which is produced from clove buds from cultivated trees. The wild clove oil contained no eugenol and deposited crystalline substances not found in ordinary clove oil.

The properties of the volatile oil distilled from the blossoms of the Indian mango tree were reported and the essential oils of several varieties of wild thyme found in Portugal were examined. Myristicin was found to be one of the main constituents of the volatile oil of harvest fennel (*Ridolfia segetum*). A sulphur compound which had not previously been described in literature was found in the oil distilled from the leaves of the sea-buchu, a South African shrub. The compound was identified as butyl-pentenyl disulphide.

Two paraffin hydrocarbons, heptane and undecane, were identified in the essential oil from the oleoresin of Coulter pine. Phellandrene and terpene hydrocarbons in the same oleoresin is a rarity.

Several investigations relative to the composition of the animal fixatives were reported. A pyridine derivative was isolated from natural musk and several additional phenols and acids were identified in castoreum. Progress was made on the problem of the

structure of ambrein, a crystalline material found in ambergris.

In the field of organic synthesis, a new group of perfume materials having intense floral odours was discovered in the ethers derived from dicyclopentadiene. Various epoxides of the ionones were prepared by treating them with hydrogen peroxide or perphthalic acid. Thio ethers having marked insecticidal properties were obtained by the reaction of saffrole and isosaffrole with mercaptans.

A study of the isomeric forms of the Grignard reagent derived from pinene hydrochloride provided an explanation for the fact that reactions involving this compound often give unexpected products. A simple method was devised for converting the reagent entirely to the form giving the desired product.

The statistical section of the report is also of considerable interest for this year as data not accessible for 1945 or the war years generally was now available. Figures appear on Italian exports, the patchouly trade in Malaya, and Taiwan camphor production, to mention only a few.

**Practical Applications of Recent Lac Research** (Revised & Enlarged Edition), Edited by H. K. Sen & M. Venugopalan (Orient Longmans Ltd.), pp. 123. Price Rs. 6/8.

INDIA PRACTICALLY HOLDS A WORLD MONOPOLY for the most versatile natural resin, lac, the cultivation and refinement of which is an important cottage industry in the country; but it is left to the advanced countries of the West to put it into use in several important industries. When the position of lac was, therefore, seriously threatened by the development of innumerable synthetic plastics, the Institute at Ranchi was established to safeguard the industry by means of research tending to make lac not only more fit for existing industries but also to find new uses for it by suitable modifications of its properties. What now remains to be done is to reduce the dependence of this natural product on foreign markets by starting such consuming industries in this country as have been made possible by the valuable researches of the Institute in this direction.

In the revised edition of "Practical Applications" under review, new chapters

have been added on ethers and ether-esters of shellac, dewaxed lac and varnish, water varnishes, instantaneous sound-recording discs and the manufacture of oil-cloth. Of the other chapters, that on hard lac resin has been expanded to include new ways of preparing the resin. Chapter VI on shellac plastics now includes a section on the possibility of manufacturing fillers in this country and chapter VI is expanded to include shellac cements, waterproof abrasive cloth and paper and possible uses of shellac in the manufacture of ply-wood, laminated boards and abrasive wheels, to mention only a few instances of enlargement of the scope of the book. Undoubtedly, therefore, the publication is of great practical importance to the industrial chemist.

The costing data, however, based on 4 annas per pound of shellac, Re. 1 per gallon of spirit and Rs. 2 per gallon of linseed oil, to take a few instances, tend to make the industrial possibilities appear too optimistic and unreal to a hard-boiled industrialist. The editors, no doubt, say in the preface that production costs are only pointers to normal conditions, but when those "normal" conditions return and whether they will return at all are highly problematical. The Institute would have done well to show in this new edition that with the prevailing prices of raw materials, the commercialization of these researches was a practical proposition.

A few slips in the book as, for example, not mentioning filtration of lac solution after treatment with activated carbon (page 11), omitting to mention the daily or monthly turn-over on which overhead is based (pages 2, 10 and 19), the unusual basis of ten working hours per day (page 28), and 55° barometer for perhaps 55° Bé. (page 82), may be corrected in the next edition. The printers, also, might have taken more care to see to the proper alignment of types on certain pages. These are, however, very minor points which do not in any way detract the value of the publication.

The book should be recommended as a very valuable technical handbook, and if only the industrialists of this country seriously consider the possibilities of establishing even a few of the industries recommended in the book, no doubt the Institute would feel highly rewarded for its efforts.

M. R.

**Centrifugal and Other Rotodynamic Pumps**, by Herbert Addison (Chapman & Hall Ltd.), 1948, pp. x+492. Price 36s.

THE BOOK UNDER REVIEW DEALS WITH CENTRIFUGAL and other rotary types of pumps which the author calls by the name of rotodynamic pumps. It is a valuable addition to the library of books dealing with centrifugal pumps. Though there are many books dealing with the design and construction of centrifugal pumps, yet Mr. Addison's book is the first of its kind in which the other types of rotary pumps like the semi-axial, axial, bore-hole and propeller types of pumps are fully described. His nomenclature of rotodynamic pumps seems to be very apt in that all the above-mentioned pumps have to be directly coupled with a rotary motive unit.

The division of the book into Principles, Design and Construction, Performance and Installations, and Operation is very logical. Though there have been repetitions of the subject-matter in more than one chapter, such repetitions have enabled the understanding of the particular problem of a pump under design, construction and maintenance better.

The chapter dealing with the design and construction is very illuminating for a student of engineering and a practising engineer. The various types of centrifugal and other rotodynamic pumps are classified and their particular merit for specific purposes are fully explained. This chapter recommends the book for inclusion as a handbook on pumps for the use of a practising engineer. The enumeration of certain practical difficulties that might arise in the handling of hot, impure and corrosive liquids is very useful. Some details of construction in handling these types of liquids, like the types of sealing rings in article 84, water-seal glands in article 87 and details of casings in article 89 add greater practical utility to the book.

In the chapter dealing with mixed flow and axial flow pumps, the reference to airfoil theory has brought the subject-matter in level with modern development. The chapter on special-duty pumps is a valuable collection of special types of pumps which a practising engineer needs most in a reference book of this type. The articles on bore-well pumps of different types with the accompanying diagrams are very well written.

Under the heading of pumps for hot and volatile liquids, the particular problems that might arise due to expansion, leakage, extreme heat resulting in the needful protection for the stuffing boxes, protection to packing suggesting their solution are useful additions to the knowledge of the working of the pumps of this type.

In the chapter on definitions and terminology, the author has clearly defined the manometric, hydraulic and actual efficiencies under different conditions of pumping. In the other two chapters following this, the author has dealt with the various causes that contribute to the realization of lower efficiencies than the designed one in actual practice and how best the gap between the two could be minimized.

The chapter on "Installations and Operations" does not form part of the theory of centrifugal and rotodynamic pumps but the enumeration of the allied machinery and auxiliary appliances has added to the usefulness of the book to the student as well as the engineer. It gives an idea of how and when a particular type of motive unit should be selected for a particular type of pump.

In the final survey of complete pumping plants, the author has selected typical examples of a simple layout, a low-lift plant, a land drainage unit, a screw type of plant, a propeller type of installation, a semi-axial and axial pump unit, vertical pumps of high-lift and low-lift varieties as in land drainage and sewage pumping and bore-hole pumping plants. The operation of pumping units in series and in parallel have been very well described with adequate sketches. The principles of boosting pumps and arrangement of boosting stations are also valuable additions. The problems of surge and slum pressures in pumping installations has been particularly dealt with in detail and solutions indicated. A few articles on mobile pumping units such as suction dredger and pontoon pumps are usefully added in the chapter on "Particular Installations".

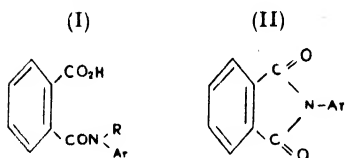
The last chapter of the book deals with certain worked examples of design, selection of pumps and auxiliary units which guide the student and the practising engineer in solving their problems.

On the whole, the book is highly commended as a text-book as well as a book of reference on the subject of centrifugal and rotodynamic pumps.

# NOTES & NEWS

## Plant Growth Regulators

A NEW GROUP OF PLANT GROWTH regulating chemicals, the *n*-aryl phthalamic acids, are described (*Science*, 1949, **109**, 588). These compounds, which are readily prepared from phthalic anhydride and aromatic amines, can be represented by the general formula (I). The esters, amides and soluble salts of these acids as well as the *n*-aryl phthalimides (II) possess similar activity.



Tomato plants having 1 to 3 unset blossom clusters were sprayed with a solution (100 c.c.) containing 100 mg. of the chemical and a wetting agent. A general pattern of response was evident. At low concentrations, the fruit set was stimulated and seedless tomatoes were produced. A morphological response involving a broadening of the leaves was also evident. As the concentration of the chemical increased, the formative effect became more pronounced. At higher concentrations (2,000 p.p.m.), the plants were definitely injured.

*N*-1 naphthyl phthalamic acid, at a concentration of 0.1 p.p.m., caused leaf roll, at 0.31 p.p.m., epinasty, and at 20 p.p.m., stem swelling. Above this concentration fruit set was completely inhibited.

Maleic hydrazide is another interesting compound which has been found to produce a pronounced, but temporary, inhibiting effect on plant growth. The effect is secured with little visible harm to the plants.

Maleic hydrazide, its cupric salt and its zinc salt, dissolved or suspended in water at a concentration of 2,000 p.p.m. when sprayed on 6" tomato plants, the plants failed to grow for a period of about 2 months. Root growth was also inhibited. After about 1 month slight chlorosis was observed. After the quiescent period, growth resumed mainly from

lateral buds. A 1,000 p.p.m. solution produced a formative effect when regrowth started from the terminal bud. Similar results were obtained using triethanolammonium and diethanolammonium salts of maleic hydrazide. These salts seemed to be more effective than the free compound. Turf was treated with 8, 4, 2 and 1 lb. per acre of maleic hydrazide. The chemical with a dispersing agent was applied as a spray in 200 gal. of water per acre. The 1 lb. rate had a slight retarding effect; the 2 lb. rate inhibited growth for 1 to 2 weeks; the 4 lb. rate for about 2 weeks, and the 8 lb. rate for over 2 months. After the quiescent period, growth appeared normal.

The chemicals have no effect on the extent or time of germination, but the seedlings suffered a marked retardation of growth.

## Synthesis of Radioactive Silk

THE POSSIBILITY OF BIOLOGICAL synthesis of a stable, homogeneous, radioactive silk protein has been discussed (*Science*, 1949, **109**, 625).

This is suggested by the unusual properties of fibroin, the protein of silk, containing  $C^{14}$ -labelled glycine and alanine in exceptionally small concentrations.

To test the ability of the silk worm *Platysamia cecropia* to incorporate radioactive glycine and alanine into the silk it synthesizes, 0.05 c.c. of a mixture of the two was injected into the body cavity of the mature silkworm (animal A). The injection contained 0.013 mg. of  $C^{14}$ -carboxyl-labelled glycine with an activity of 18,000 cpm. and 0.02 mg. of  $C^{14}$ -carboxyl-labelled *dl*-alanine with an activity of 9,000 cpm. (Geiger Muller).

A thin piece of cocoon fabric spun by the worm after 24 hr. of normal feeding exhibited radioactivity. A similar result was obtained with a silk-worm (animal B) injected with 0.05 c.c. of a solution containing 0.04 mg. of  $C^{14}$ -carboxyl-labelled *dl*-alanine alone, with an activity of 18,000 cpm. Fragments of cocoon from each insect placed under an unexposed film and later developed, invariably gave radioautographs

revealing details of the pattern of the silk fibres.

To test whether radioactivity of the silk was actually present in the component amino acids, the protein hydrolysate was subjected to ninhydrin procedure. Duplicate counts of the barium carbonate precipitate revealed 160 and 150 cpm. per 0.1 mm. barium carbonate, thus showing the radioactivity to be present in the  $\alpha$ -carboxyl group of some amino acid associated with the silk fibres.

To make sure that the labelled amino acids were actually incorporated into the peptide chain structure of the protein itself and that they were not adsorbed into the silk fibres, reprecipitated protein was tested for radioactivity. The hydrolysate from 13 gm. of this protein, on ninhydrin determination, gave 12.7 cpm. per 0.1 mm. barium carbonate, or 127 cpm. per 0.1 mm. of hydrolysate of labelled silk fibroin as corrected to standard conditions.

The above results led to experiments on protein synthesis in the isolated silk gland of the insect. Four silk glands from two mature insects were incubated at 37°C. in an atmosphere of oxygen. The proteins from the glands, gave on assay for radioactivity, a total of 74 cpm. in the 0.067 mm. sample of barium carbonate or 93 cpm. per 0.1 mm. of barium carbonate as corrected to standard conditions, implying thereby that the amino acids were incorporated into protein in the living silk-worm gland *in vitro*. Since the fibroin was not isolated, there is no certainty, however, that silk rather than some other protein was synthesized under the conditions of the experiment.

There is the possibility that the labelled amino acids may have entered preformed fibroin molecules by "exchange" rather than by participation in a true synthesis of the fibroin molecule *de novo*. There may also be the formation of side-chain peptide bonds between the labelled amino acids and either amino or carboxyl groups not in the  $\alpha$ -position.

## Storing Seed Potatoes

STORING OF SEED POTATOES in artificially lighted cellars to check their sprouting and consequent loss of nutrient under ordinary storage conditions is discussed (*Philips Tech. Rev.*, 1949, **10**, 318).

Sprouting is checked at low temperatures (2° to 4°C.), and with a little light at 5° to 9°C. While

the former method is neither practicable nor economically justifiable, the latter involves tedious temperature regulations in the frosted glass-sheds where the potatoes have to be stored.

The effects of artificial light on sprouting were, therefore, investigated. One part of an underground test cellar with potatoes so stored in shallow boxes as to allow light to penetrate between them was illuminated with ordinary incandescent lamps and another part with fluorescent lamps of the "daylight" colour. The lighting was on continuously for a period of 3 months after which period the potatoes were taken out for planting.

In the first part lighted with 6 incandescent lamps installed in each section of  $7 \times 8$  m., placed at intervals of 3 m., potatoes were found in a better condition than those which had been kept in clamps, but sprouting was not well checked. Firstly, the potatoes in the immediate vicinity of the lamps sprouted strongly notwithstanding the fact that they were receiving a fair amount of light. This is explained by heat radiations from the lamps which helped sprouting. Secondly, light being radiated from a central point in the incandescent lamp, there was little uniformity in the radiation. Large spaces were, therefore, left without any light.

The second part of the cellar illuminated with 5 TL fluorescent lamps of 40 w. installed in each section of  $7 \times 8$  m. gave better results. In this case radiation of light was uniform all through and there were no shadow points, the lamps being mounted vertically against the wall. Moreover, there was scarcely any heat radiation to promote sprouting.

Investigations were then made on the kind of light which checks sprouting most and to ascertain what amount of light—using light of a certain wavelength—was just capable of sufficiently checking sprouting.

One lot of potatoes was irradiated with various intensities of blue and another lot with different intensities of red light. For the first lot a TL lamp was used with magnesium tungstate as lumino-phore and the addition of a blue filter; for the second lot a lamp with cadmium borate was used, with a red filter. The irradiation took place at  $14^{\circ}\text{C}$ . It was found that blue-violet light strongly checked the growth of sprouts, while red rays had but little effect.

Very little research having been done into the mechanism of sprouting of potatoes, it was presumed that the blue-violet light destroys the auxin—a hormone which is responsible for growth of sprouts.

Further irradiation tests were so arranged that the light fell continuously upon the potatoes from one side (as would be the case in the storing of potatoes).

Under blue light of an intensity up to about 30 erg./sq. cm. sec., the sprouts were perceptibly checked in growth. The sprout that had received about 2 erg./sq. cm. sec. was still somewhat phototropically directed.

Potatoes under red light, however, showed an unexpected phenomenon. Even a much lower luminous intensity of 1 erg./sq. cm. sec. (than with blue light) produced noticeable results. Moreover, the checked sprouts did not grow towards the light, not even under much stronger radiations.

From these experiments it appears that the checking of sprouting—at least under the influence of red light—is not to be accounted for by reason of a growth-stimulating substance being rendered inactive by the light. Rather it is to be supposed that, under red light, a growth-checking substance in the potatoes is activated. The question whether this hypothesis of activation of a growth-checking substance is correct is to be further investigated experimentally.

### Continuous Superphosphate Process

A NEW SUPERPHOSPHATE PROCESS with centralized control known as the "Sokett Super Flo-process" is described (*C.T.J.*, 1949, 125, 103).

The raw materials, Florida pebble rock and  $66^{\circ}\text{Bé}$ . sulphuric acid are treated in an acidulating tower into which reactants are introduced in a finely divided state: the ground rock as a dust stream and the acid as an atomized spray. The reaction is very rapid. The superphosphate has a hard porous grain structure and possesses good physical properties for ammoniation, handling and application.

The rock, ground to 100 mesh in an air-swept Raymond pulverizer, is conveyed to continuous weighing machine by screw conveyor and bucket elevator. An air seal between the pulverized rock-feeding spout and the machine makes continuous weighing

possible by checking the fluid flow of the rock. For average operation, the scales are set to weigh out 25 tons of ground rock per hr. which, on reaction with approx. 20 tons of acid per hr., constitute a 45 ton per hr. operation.

The weighed rock dust is introduced through a horizontal steel air duct tangentially into the upper side of the cylindrical rubber-lined steel acidulating tower. Acid at  $115^{\circ}$  to  $135^{\circ}\text{F}$ . is delivered to the tower at the rate of 20 tons per hr. by a stainless steel atomizing nozzle in the centre of the tower.

The tower discharges directly into a puddler over which it is situated. The puddler has a rubber-lined housing and is equipped with propelling flights which pass the viscous material along to solidifier. The solidifier, a channel-like pallet conveyor made up of steel plates formed in a U-shape, fulfils the function of the den in the order process, the material leaving it being solid superphosphate ready for storage and curing.

The centralized control of the process makes it possible for a 3-man crew to operate a 45-ton per hr. plant as against 10 to 12 crew required with a den plant of about equal capacity. Power costs also compare favourably, the Super Flo-process using 1.12 kW.-hr. per shrunk ton as compared to 1.75 kW.-hr. in the den installation.

### Beneficiation of Lead-zinc Ore from Zawar Mine

THE LEAD-ZINC MINE OF ZAWAR is situated about 25 miles by road from Udaipur, Rajputana. Two picked samples of ore from the mine have been investigated and the results of the tests have been recently reported (*Trans. Geo. Met. Inst. of India*, 1949, 45, No. 1).

The first ore sample (—100 mesh) analysed to the following:

		%
Pb	...	14.10
Zn	...	6.97
Fe	...	2.67
Insol.	...	41.52
CaO	...	9.60
MgO	...	5.74
S	...	6.23
Sb	...	0.064
Cd	...	0.059
$\text{Al}_2\text{O}_3$	...	0.21

A second ore sample investigated had an assay value of 0.20 per cent lead and 5.3 per cent zinc.

Concentration of the ore by gravity methods—jigging and tabling—and by flotation, was tried. In the case of the second ore sample, the flotation tests indicated that the lead, although present in low amounts, could be successfully recovered as a lead concentrate of reasonably good grade.

On the basis of the experimental work on ore samples containing 0.2 to 2.0 per cent lead and 4.0 to 10.0 per cent zinc, a flow sheet for a pilot mill to treat 6 tons of ore per hour has been drawn up and machinery for such a plant has been specified. Recoveries from this plant could be expected to exceed 90 per cent of the lead in a concentrate assaying lead, 66 per cent; zinc, 6 per cent; and 85 per cent of the zinc in a concentrate assaying zinc, about 63 per cent and lead, less than 0.5 per cent.

The silver content of the lead concentrates is so low (approx. 6.5 oz./ton) that recovery of silver would not be justified.

The cadmium-content of the zinc concentrates is such that if electrolytic methods of zinc production were adopted, a valuable cadmium by-product could be obtained.

### De-enamelling

THE SALVAGING OF PORCELAIN enamel parts, not suitable for repair, by de-enamelling is described (*Amer. Cer. Soc. Bull.*, 1949, 28, 183). The process can be applied if it is possible to salvage the formed-metal shapes involved at a cost less than, or equal to, the metal shapes involved, fabricating cost, and if the salvaged metal is suitable for re-enamelling. Naturally, in times when metal shortages are involved, the salvaging of parts to maintain production operations will considerably outweigh many of the cost considerations. Additional factors involved are concerned with the cause of final rejection of the part in question, that is whether the fault is due to a flaw or failure in the part itself which is not repairable, or whether it is due to failure in the enamelling operation. There is no economic advantage to de-enamel parts which cannot be salvaged.

Two methods are employed: (1) chemical solution; and (2) mechanical removal. In many cases, de-enamelling procedures combine both methods. For the chemical dissolution of enamels,

the following procedures are employed: (1) acid de-enamelling employing hydrochloric, sulphuric, acetic and hydrofluoric acids, and combinations thereof; (2) the use of caustic alkalies, either molten or in boiling water solutions. In some cases combinations of acid and alkali treatments have been employed.

Of the mechanical means employed, the most satisfactory has been the use of sand-blast.

In one method employing sulphuric acid, the ware was placed in a tank containing acid at a temperature of 140° to 150°F., held at this temperature for 1 hr. and then allowed to cool. De-enamelling was reported to be complete after 12 hr. A recent process consists in immersing the ware to be de-enamelled in a 10 per cent sulphuric acid solution with 0.03 per cent of a commercial inhibitor. The acid is held at 180°F. and the ware remains in the tank for 30 to 50 min. Following this operation, the ware is rinsed in a hot-water spray, neutralized in a second spray, and dried and sand-blasted to remove sludge and any enamel which has not been taken off. This procedure appears to hold considerable promise for de-enamelling non-acid-resistant coating.

In the process employing molten caustic soda (*U.S. Patent*, 528,156) the ware is de-enamelled in a large heavy-walled steel tank, electrically heated and filled with molten caustic soda. The process is rapid during the first hour or two, de-enamelling being complete in a few minutes; but after 5 or 6 hr., the time required increases. So, it is customary to stop the operation after 8 hr. and allow the sludge to settle. Improvements incorporated in the procedure are: (1) the caustic bath is heated by electrical resistance, the current being passed through the bath; and (2) caustic soda is mixed with other ingredients which help to control the flow current and prolong the life of the bath.

The method is expensive and the removal of adhering caustic soda is difficult. No lead enamels can be used in this process.

A boiling solution of caustic soda (5 to 15 lb. per gal. of water) has found the most general use in de-enamelling processes. The ware is immersed in the boiling solution for 10 to 12 hr. during which period either the ware is agitated or the solution is circulated. Following de-enamelling, the ware is normally cleaned by high-

pressure spray, in some cases followed by wire brushing or light sand-blasting. This method has not found much favour as it is often difficult to produce first-quality ware on re-operation.

Sand-blasting usually employed to remove porcelain enamel from cast-iron surfaces has several advantages. It provides a very high-quality surface for the re-enamelling operation, eliminating the possibility of nickel-flaking, caustic carry-over and blistering which have been the major defects encountered with chemically de-enamelled ware. It has been found that by controlling the orifice size, air pressure, and the angle of incidence of the blast, it is possible to sand-blast ware of 24 gauge thickness without undue difficulty. When the blast is directed at an angle of 15° to 20° from the horizontal, warping of the ware is eliminated. Distortion is further minimized by working across the piece from front to back so that there is always a coating of enamel ahead of the sand stream.

### Properties of Some Indian China Clays

INVESTIGATION OF THE MINEROLOGICAL and colloidal nature of 4 important Indian China clays, viz. Rajmahal clay, Kasimbazar clay, Travancore clay and Banda clay, by chemical analysis, electro-dialysis, viscosity measurements, differential thermal analysis and examination by X-ray and the petrographic microscope, has been recently completed (*Amer. Cer. Soc. Bull.*, 1949, 28, 187). The results of this investigation show that these clays are essentially kaolinite and had largely the properties of typical China clay.

Tables I and II give the chemical analyses of the untreated and electro-dialysed clays.

The theoretical mineral constitution of these clays, as calculated from the chemical analyses of the untreated clay, is given in Table III.

The morin dye test was used to detect the presence of free aluminium ions in the clear water extract of the clays and it can be used as an indirect measure of stability. The clay is leached by soaking in dilute hydrochloric acid overnight and then taking the supernatant liquid for test. The liquid is neutralized with caustic soda and then made slightly acidic. On addition of a few drops of morin dye, a greenish-yellow fluorescence is observed under



ultra-violet light if aluminium ions are present. The greater the intensity of the fluorescence, the greater the number of free aluminium ions in the test solution. The dye test shows that Kasimbazar clay is the least stable, Banda and Travancore clays are slightly more stable and Rajmahal clay is still more stable in so far as it loses its aluminium ions with difficulty.

Thermal analysis of the clays showed that these clays consist mostly of kaolinite. The Banda and Travancore clays contain larger percentages of kaolinite as their intensity in reaction is greatest. The Rajmahal clay comes next and the Kasimbazar clay shows the least intensity, thereby showing the presence of considerable amounts of other minerals.

Both untreated and electro-dialysed clays were examined by the powder method in a General Electric XRD X-ray unit. The  $d$  values of these clays were compared with the  $d$  values of the Langley clay from North Carolina

which was taken as the standard kaolinite. Except Kasimbazar clay, the others gave peaks at the same angles and almost the same intensity as the standard. Quartz, feldspar, mica, etc., usually associated with the clays, especially with kaolinite, were not detected in the three clays. Kasimbazar clay, in addition to kaolinite peaks, gave peaks for other minerals, particularly muscovite.

Petrographic examination was conducted on crushed clays (100 mesh). The results showed that Rajmahal clay was fine grained, and very few hexagonal kaolinite crystals were seen at high magnification. Grains of quartz were present. The heavy residue consisted of clear, rounded quartz, with a few flakes of muscovite and very few grains of magnetite. Banda clay was coarser in texture. Kaolinite "shrimps" and flakes were common. Rutile, titanite, tourmaline, magnetite, zircon and quartz were found in the residues. Kasimbazar clay showed crystals of kaolinite, quartz and muscovite. The residues contained mica,

quartz and feldspar. Travancore clay was very fine grained, and it was difficult to observe single crystals although occasional typical kaolinite "shrimps" were found. The residues contained very small grains of quartz, both round and angular, and few flakes of mica.

Particle-size measurements indicate that the Travancore clay has the least material in the range above 10 microns, the Banda clay is only slightly coarser, and that the Kasimbazar clay is the coarsest of the group. Rajmahal clay has an appreciable content of relatively large grains and, at the same time, the largest amount of fine material below 1 micron. These conclusions have been confirmed by electron-microscope studies.

A study of the colloidal behaviour of the clays by viscosity and pH measurements on samples of electro-dialysed clays shows that they show little base-exchange capacity, which is characteristic of kaolinite.

### Impervious Crucibles

CRUCIBLES WITH IMPROVED STRUCTURE and highly impervious lining have been developed at the U.S. National Bureau of Standards (*Chem. Age*, 1949, 61, 13).

The new crucibles can withstand attack by highly corrosive melts on account of minute inter-connecting passages between their pores and as such can meet the exacting requirements peculiar to the production of optical glass and the smelting of highly corrosive high-lead glazes and enamels.

In the method employed, the clay casting slip—the mixture of dry ingredients with the proper amounts of deflocculants and water (to give good flow characteristics)—is poured into plaster-of-Paris moulds consisting of two parts, a shell and a core, locked in position before the pot is cast. During the initial stage of casting, the mould is slightly tilted to lessen the tendency to entrap air bubbles between the lining and the pot. After the bottom is filled, the mould is levelled and rocked lightly until the pot is completely cast.

Lining of the pots is applied before the pots are cast. In other words, the lining slip is first poured around the core of the mould, which is removed and transferred to the shell. The pot is then cast in the same manner as for unlined pots. Double and triple layers of lining, up to a

TABLE I — CHEMICAL ANALYSIS OF THE UNTREATED CLAYS

	RAJMAHAL CLAY %	KASIMBAZAR CLAY %	BANDA CLAY %	TRAVANCORE CLAY %
SiO <sub>2</sub>	54.07	47.71	43.92	44.84
Al <sub>2</sub> O <sub>3</sub>	32.10	34.93	41.12	40.70
Fe <sub>2</sub> O <sub>3</sub>	0.30	0.92	0.72	0.21
CaO	0.69	1.62	Trace	0.66
MgO	0.12	1.92	Trace	Trace
Alk.	0.35	1.10	0.53	0.20
Ignition loss	12.37	11.80	12.71	13.39
Total	100.00	100.00	100.00	100.00
SiO <sub>2</sub> ·Al <sub>2</sub> O <sub>3</sub>	3.00	2.30	1.80	1.80

TABLE II — CHEMICAL ANALYSIS OF THE ELECTRO-DIALYSED CLAYS

	RAJMAHAL CLAY	KASIMBAZAR CLAY	BANDA CLAY	TRAVANCORE CLAY
SiO <sub>2</sub>	55.44	50.21	45.36	45.61
Al <sub>2</sub> O <sub>3</sub>	30.67	35.60	41.98	40.40
Fe <sub>2</sub> O <sub>3</sub>	0.14	0.21	0.09	0.01
CaO	Nil	Trace	Nil	Trace
MgO	Nil	Trace	Nil	Trace
Alk.	Nil	Trace	Nil	Trace
Ignition loss	13.75	13.92	13.01	13.86
Total	100.00	100.00	100.00	100.00

TABLE III

MINERAL	RAJMAHAL CLAY	KASIMBAZAR CLAY	BANDA CLAY	TRAVANCORE CLAY
Orthoclase	1.7	6.1	2.72	1.1
Anorthite	3.6	7.3	...	3.3
Haematite	0.3	0.8	0.6	0.2
MgCO <sub>3</sub>	0.3	3.6	...	...
Kaolinite	78.0	76.3	89.2	92.0
Quartz	15.8	5.9	...	...
Alumina	...	...	7.5	3.3



total thickness of  $\frac{1}{4}$ " or more, are sometimes applied.

The additional step that has been introduced for lining crucibles consist in spraying a coat or coats of clay slip over the lining to seal incipient cracks. Such faults often open during use and permit a fluid glass or glaze to penetrate into the walls or, in some instances, to leak through the crucible.

The cores are removed as soon as practicable after casting in order to prevent cracking of the lining—timing of this operation is critical—and the shells are removed when the pots have set sufficiently to retain their shape. The pots are then air-dried and finally, immediately before use, are heated to approximately the temperature of the melt they are to contain. A carefully regulated heating schedule has to be followed and the rise in temperature controlled in accordance with the relative speed with which the pot can be heated through certain ranges without damage.

In addition to its unusual pore structure, the new crucible has low gas permeability (even though it shows a porosity of 22 per cent after firing at 1435°C.). The crucible has a somewhat low resistance to thermal shock.

## High-temperature

### Laboratory Kiln

AN INEXPENSIVE ALUNDUM-LINED kiln that can be fired to 1950°C. in about 40 min. with uniform temperature control is described (*Amer. Cer. Soc. Bull.*, 1949, 28, 192). The kiln is a small up-draft laboratory kiln, unique in both ease of firing and replacement of lining. Standard gas-welding equipment was found suitable as it provided high flame temperatures with a minimum of combustion space.

The interior of the kiln (9"×9"×7") is lined with standard 9" straight brick of high-purity (99 per cent), electrically fused alumina. These had been fired to cone 35 (1785°C.) in the course of manufacture. The lining was insulated with 2900°F. insulating brick. The use of high-purity alumina refractories not only allows high temperatures to be reached but permits unusual kiln atmospheres without the danger of flashing and staining caused by volatilization of impurities in furnace lining. Temperatures as high as 1950°C. were reached without lining injury. For temperatures above 1900°C., elec-

trically fused magnesia and electrically fused stabilized zirconia are suitable. Under strongly oxidizing conditions, magnesia is satisfactory. Under reducing conditions, however, there was a tendency for magnesia to volatilize at high temperatures.

Replacement of refractories in the kiln is easily accomplished as the lining is not cemented and all the parts can be readily lifted out and new posts set in. This was found to be a distinct advantage as it permits free expansion and contraction of the lining without development of cracks.

In 40 min., a temperature of 1950°C. was reached with either oxygen-propane torches equipped with multi-flame heating tips or oxygen-acetylene torches equipped with standard welding tips. The former permits slower heating schedule and more uniform temperatures. For temperatures below 1,000°C., small gas-air burners can be used.

The advantages of using propane in lieu of acetylene are numerous. With propane multi-flame tips operate more successfully. Because propane is stored as a liquid in tanks, changes are less frequent, and it is safer to store and use than acetylene. The main advantage with acetylene is its higher flame temperature.

Torches should be so placed that the flame does not impinge on the ware. The burner ports are cut in the brick and should not be more than  $\frac{1}{4}$ " larger than the burner-tip diameter. It is advisable to set the ware on refractory pedestals or trays about  $\frac{1}{4}$ " above the hearth to permit free circulation. The burner tips are placed flush with the outside surface of the inner refractory lining and concentric with the burner ports. The tips are water cooled. These precautions prolong their useful life. The tips have 2 or 3 turns of copper tubing which are silver soldered. With acetylene, welding tips having an orifice 0.067" to 0.052" are required. With propane, No. 4 multi-flame heating tips were used. The kiln has only 2 burners firing from opposite sides.

### Electric Gauge

AN ELECTRIC GAUGE HAS BEEN developed for measuring the bores of small guns or tubes of about  $\frac{1}{4}$ " inside diameter (*J. of Res.*, 1949, 42, 461).

The gauge operates on the principle of mutual inductance of two solenoids mounted on each

prong of a fork. A constant voltage is maintained in the primary coil. The current induced in the secondary is a measure of the distance between the two coils. The gauge is calibrated with rings of known diameter and the diameter of a tube or gun bore is read directly on the dial of the instrument.

The gauges so far available, though quite satisfactory for production control, consume much time, are unwieldy and are not quite accurate for research work. The electric gauge described is relatively inexpensive, quick and accurate, gives continuous reading and has a sensitivity of 0.002" which holds over the entire range of 0.02" without recalibration. A wider range than 0.02" can be obtained without recalibration by exchanging the gauge points for another pair and taking the difference in height of the points into consideration. The nearer the gauge points are situated to the Y of the fork, the greater will be the mechanical magnification of their motion by the solenoids. A variation in the use of the gauge is to connect the output to a recording potentiometer instead of to the micro-ammeter. In this way a continuous record of the profile of the bore of a tube can be obtained. This feature of the instrument has, however, not been fully developed.

### New Synthetic Fibre

FIBRES FROM COTTON-SEED PROTEIN and chemically modified cellulose produced experimentally at the U.S. Bureau of Agr. & Ind. Chemistry's Res. Lab., are described (*Chem. Age*, 1949, 61, 64).

Cotton-seed fibre is obtained by a special acid treatment of the proteins from solvent-extracted cotton-seed meal. This changes the structure of the protein, corrects its tendency to gel or lump in untreated dispersions, and makes it suitable for spinning.

The fibres from sodium carboxymethyl cellulose (a soluble compound made from wood or cotton cellulose) are derived by extruding its solution in water through a spinnerette into a bath containing the salt of one or more of heavy metals. The fibres being soluble in soapy water or other weak alkali solutions (similar to alginate fibres from alginic acid, a constituent of sea weed), are considered suitable for use as a spacing agent—to provide the "missing threads"—in specially woven fabrics.

### Drying Oil Improver

A SYNTHETIC ORGANIC CHEMICAL, "dryene", for improving naturally occurring drying oils, has been developed by *Carbide & Carbon Chemicals Corp.*, New York (*Chem. Eng.*, 1949, **56**, 172).

A low molecular weight polyester, "dryene" is a viscous liquid of low volatility. Under conditions of a varnish or alkyd cook, it undergoes ester interchange and dehydration to yield a product structurally similar to that obtained from a 6-carbon dienoic acid. For purposes of determining the balance between hydroxyl and acid groups in an alkyd formulation, the up-grader may be assumed to be an acid with a combining weight of 112.

The action of "dryene" is based on the introduction of increased conjugated unsaturation into a finish formulated from a soft-drying oil, permitting a high degree of cross-linking by the formation of carbon-to-carbon linkages rather than ester linkages, the former being more stable than carbon to oxygen linkage, typical of phthalic anhydride-glycerol alkyls.

The improved drying characteristics of the oil reacted with the up-grader make it of great interest in treating oils to produce alkyd resin varnishes with improved alkali and water resistance, more rapid set, improved mar resistance and increased toughness and durability. The same results are obtained by incorporating "dryene" in varnishes and alkyd resins.

### Modified Drying Oils

MODIFIED OILS HAVING SPECIAL setting properties have been prepared by reacting soya-bean and linseed oils with dicyclopentadiene (*Chem. Eng.*, 1949, **56**, 186).

Dicyclopentadiene is depolymerized by heat and the monomer reacts with the double bonds of the oil in accordance with the Diels-Alder reaction. Linseed and soya-bean bases have been called "Cykelin" and "Cykelsoy" respectively.

Both the products have practically the same properties and are used for similar purposes except that the linseed base has lighter colour and sets quicker than the soya-bean base. Both set in a few hours, dry hard overnight, give films resistant to water and alkali, and are miscible with bodied or unbodied oils. Their use is specially recommended in varnishes and enamel vehicles.

### Foam Inhibitors

THE TENDENCY OF LUBRICATING oils to foam in use, which reduces lubricating efficiency and impedes oil circulation in I.C. engines has been studied. Recycling of the oil through a column of activated charcoal at 50°C. or lower, reduced foam to  $\frac{1}{10}$ th the volume of the foam from untreated oil. Activated manganese dioxide, silica gel, and "porocell", when packed in columns for recycling, also defoam the oil to a lesser extent (*Chem. Age*, 1949, **61**, 47).

### Insect-proofing of Cotton Flour Bags

DISCOVERY OF A NEW TREATMENT to make cotton flour bags insect-proof without impairing the quality of the flour is claimed by *U.S. Dept. of Agriculture*. The most effective treatment is stated to consist in spraying 10 mg. of pyrethrins alone or mixed with 100 mg. of piperonyl butoxide per sq. ft. of cloth. Bags so treated admitted no insects during seven months of exposure to *Cadelle*, the Mediterranean flour moth (*USIS*).

### Wood Preservative

A NEW CHEMICAL WOOD PRESERVATIVE, copperized chromated-zinc chloride, developed by *Koppers & Du Pont*, is described (*Chem. Eng.*, 1949, **56**, 186).

Copperized CZC containing 73 per cent zinc chloride, 20 per cent sodium bichromate, and 7 per cent cupric chloride possesses greater permanence of its toxic constituents than CZC. The metal corrosion factor, the glow characteristics, the handling and control, however, of the two preservatives are reported to be about the same.

### Electronic Filing Machine

AN ELECTRONIC MACHINE THAT can file away documents on 10,000,000 different subjects, then automatically select documents relating to one subject and make a picture record of them in a few minutes, was recently demonstrated at *U.S. Dept. of Agriculture*, Washington.

The new machine, called the "Microfilin Rapid Selectro", combines the actions of a motion-picture project photo-electric eye, and a high-speed camera.

Documents to be filed in the machine are first micro-filmed on conventional 35 mm. film. The

contents of 50,000 ordinary library cards can be put on a single reel of film.

While a document is being micro-filmed, a code pattern, consisting of black and white squares, is filmed beside it to indicate the subject to which the document relates.

To operate the machines, a master code card is inserted into a slot in the machine. On the master card is a code pattern identical with that appearing besides all micro-filmed documents relating to a subject, say penicillin. As a reel of micro-film documents passes rapidly through the machine, a photo-electric eye scans them, and when a document having a code pattern similar to that of the master code card for penicillin appears, it breaks an electronic beam, tripping the shutter of a super-fast camera. The camera instantaneously takes a picture of the document. (*USIS*).

### New Colour-computing Device

A NEW DEVICE FOR MEASURING and analysing a large number of colours with precision and rapidity known as General Aniline-Libroscope Tristimulus Integrator is described (*J. Franklin Inst.*, 1949, **247**, 541).

The integrator, an electronically controlled ball and disc mechanism, integrates the three components of colour. First the light, which is energy radiating from the sun or the lamp, secondly the dyed or coloured object under observation which reflects varying percentages of light of different wavelengths, and finally the eye itself. Internationally accepted data on light and the eye are available and reflectance of the object is measured by a spectrophotometer. The mathematical combination of these factors, hitherto involving multiplication at scores of wavelengths and addition of the products, is achieved in 2.5 min.

### Improved Plough

THE RECENTLY INVENTED DOUBLE plough consisting of double bottoms of two standard *desi* ploughs (suitably coupled by means of an iron framework and pulled by a single central beam) has been further modified by the *Indian Agricultural Research Institute*. The new plough can tackle with ease the most difficult fields infested with weeds or left with stubbles of previous crops.

### Ciba Science Centre

*Ciba Ltd.*, of BASLE, SWITZERLAND, have established a unique institution in London, the Ciba Foundation for the Promotion of International Co-operation in Medical and Chemical Research. The Foundation will hold informal conferences from time to time to which scientists will be invited, and lecturers from abroad will be asked to address. Besides, grants will be made to younger men of outstanding merit and enable them to study methods of research. The trustees will provide accommodation for other scientific bodies and universities in the buildings, for purposes within the objects of the Foundation (*Chem. Age*, 1949, 60, 915).

### Indian Standard for Plywood Tea-chests

INDIAN STANDARD SPECIFICATION for Plywood Tea-chests lays down the definitions used in the plywood industry and trade, standard specifications for plywood and dimensional standards for tea-chests and components, and test methods for plywood for tea-chests.

Of the seven appendices included in this standard, the first one catalogues the various timbers that are to be used for plywood and the treatment to be given to them in the manufacture of plywood for tea-chests. The rest of the appendices detail the various methods of test connected with this standard and the form of report of results of tests. A useful table, laying down the dimensions of component parts for tea-chests of various sizes, is included.

The publication is priced at Rs. 1/12. Copies can be had from the Secretary (Administration), *Indian Standards Institution*, Old Secretariat, Delhi 2.

### Wool Grading

STANDARDS FOR GRADING OF Indian wool have been drawn up by the Textile Division Council of the *Indian Standards Institution*. The step will enable the Indian wool-exporting industry to solve problems arising from the vari-

ability and the uncontrolled quality of wool that is being sent out. The standard apply to wool produced in India and specify grades according to colour and quality. Specifications for packing materials are also included.

The publication is priced at As. 8 and can be had from the Secretary (Administration), *Indian Standards Institution*, Old Secretariat, Delhi 2.

### Announcements

*Australian Fellowships for Indian Students* — Australia has offered 5 scholarships for the year 1950 to Indian students for higher training in industry and technology and postgraduate studies in Australia.

*UNESCO Fellowships* — India has received during 1947-48, 18 fellowships — 15 awarded by U.N. and 3 by UNESCO — for studies in social welfare, health work, and physical and biological sciences.

*An International Institute for Research in Sound* has been established in Paris to co-ordinate world research in acoustics. For the time being, the Institute will concentrate on problems such as design of concert halls, distribution of orchestral instruments and fight against noise in large cities.

### REPORTS FROM STATES & PROVINCES

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heavy rains would preclude composting operations) is expected to benefit an area of 10,000 acres each year.

#### Thermal Station at Kurnool

A SCHEME FOR THE INSTALLATION of a central thermal station at Kurnool at a cost of Rs. 145.27 lakhs has been sanctioned. The station will have two 5,000 kW. generating sets with sub-stations at Dhane, Gooty, Pamidi and Anantapur and will cater to power requirements of important industries being planned in and around Kurnool and also provide scope for large-scale rural development.

### NEW DELHI

#### Wheat Rust Control

A COMPREHENSIVE SCHEME FOR research on an all-India basis aiming at evolving a type of wheat which is simultaneously resistant to all the various kinds of rust — keeping in view the botanical, mycological and agronomical aspects of the problem — has been sanctioned by the Central Ministry of Agriculture. A sum of Rs. 8,58,000 has been sanctioned for the purpose for the first 5 years.

So far, research has been concentrated mostly in finding out the physiology and etiology of the 3 kinds of rust — black, brown and yellow. The Wheat Rust Control Committee, appointed in 1947, recommended trial, as a short-term scheme, of the theory that while infection is killed in the plains by intense summer heat, it is carried from season to season by the summer sowings of wheat and barley mainly in the Nilgiris and Palanay Hills. On this basis, it is held that if sowings of wheat and barley in summer are completely stopped in Peninsular India, the source of infection will disappear and the menace of rust will be mitigated if not completely eradicated. This theory is being given a trial in consultation with provincial and state governments concerned.

Seed-mixture trial is the second short-term scheme recommended by the Committee and aims at mixture of those varieties of seeds which are claimed to be tolerably resistant to the 3 kinds of rust individually. This scheme is now a part of the Grow More Food Campaign.

Under the long-term scheme now sanctioned, research would be conducted on the following lines: (a) finding out physiological races of the 3 kinds of rust and maintaining their live culture; (b) breeding of wheat varieties resistant to all the 3 types of rust; and (c) ascertaining whether the effects of rusts can be minimized by changing cultural and manurial practices, e.g. cropping, spacing and so forth.

# Reports from States & Provinces

## MYSORE

### Plans for Development of Technical Education

A CONFERENCE PRESIDED OVER BY the Dewan of Mysore and attended by the heads of technical institutions and other state authorities was recently held in Bangalore to discuss ways and means of developing technical education in the State.

The conference first considered the question of co-ordinating all technical education in the State and steps to be taken for avoiding duplication of instruction in various institutions. The conference emphasized the need for practical training in the various courses of study and the need for further applied training for students in industrial establishments before the diploma is given.

The conference recommended the establishment of an advisory body, the Board of Technical Education, with the following functions:

(i) To review from time to time the courses of studies and practical instructions given in various technical institutes;

(ii) to suggest alterations or modifications in such instruction;

(iii) to propose courses of study and instructions in the new technical institutes that may be established;

(iv) to advise on the opening of new technical institutes at any centre in the State; and

(v) generally to advise on all matters connected with technical education through such institutes.

The appointment of a Director of Technical Education to supervise the working of the various institutes and to advise the Board and the Government on the co-ordination of the activities of these institutes was recommended by the conference.

The need for establishing a school of arts and crafts for imparting instruction in fine arts was confirmed by the conference. It was suggested that the Chamarajendra Technical Institute at Mysore may form the nucleus of such an institute.

The conference also recommended that when all the technical institutes are functioning, a Central Technical Examination Board may be formed.

## RAJASTHAN UNION

### Tapping Sub-soil Water

POSSIBILITY OF FINDING SUB-SOIL water in Samdari, a small town in Marwar, 50 miles west of Jodhpur, is being explored by irrigation engineers of the Government of India.

Samdari, situated at the apex of the Luni river in its depression area. From investigations already carried out, the sponsors of the scheme hope that in a 40-mile stretch of land, beginning from Samdari to Tilwara, fresh water exists which, at a conservative estimate, would be able to irrigate immediately at least 8,000 acres of land besides providing sweet water to thousands of people in this area.

Explorations made at a spot 3 miles from Samdari railway station resulted in a find of water (6 ft. from the surface) whose level was unaffected even after pumping out for several hours at 40,000 gal. per hr. indicating thereby that the rate of infiltration from the sub-soil was equivalent to the rate of pumping.

Samples of water from this area have been found sweet. 20 miles down-stream from Samdari, water becomes slightly brackish although it is considered good enough for cultivation. Besides the saline content of water is considerably reduced during and after the rains as rain water dilutes the salts, making cultivation of *rabi* crops possible.

On the question of water supply depends the development of this fertile area for agricultural purposes. Water will also make afforestation possible so that passing monsoon from the Arabian Sea is attracted, thus increasing the annual rainfall. The investigations now proceeding are, therefore, of vital importance to Marwar.

## BOMBAY

### Potato Cultivation

TO AUGMENT THE PRODUCTION OF potatoes in the province, the Government is supplying cultivators 131,000 bags of seed potatoes, 5,000 tons of powdered groundnut oil cake and 87,000 bags of special manure mixture during the *rabi* season and 35,000 bags of seed potatoes and 4,300 tons of powdered groundnut oil cake during the 1950 *kharif* season.

A committee of 8 non-official representatives will arrange procurement and despatch of seed potatoes from Simla Hills during the season.

## EAST PUNJAB

### Improved Types of Wheat & Barley

THE EAST PUNJAB AGRICULTURE Department has evolved 2 improved types of wheat and 1 of barley which are fairly resistant to rust and are high yielders. One variety of wheat, No. 3970, yields 1.8 md. to 2.2 md. more than the standard varieties, No. C.591 and C.250. This variety is also fairly resistant to all the 3 types of rust. The other high-yielding type yields 28.4 md. of grain per acre as compared with 23.15 md. by C.591.

The new variety of barley evolved possesses bright and plump grain and gives higher yields than the common barley varieties, T.4 and T.5. This variety, which is resistant to loose-must disease, is of a good malting quality. Its yield per acre in irrigated areas is 41.3 md. as against 38.6 md. of the T.4 variety.

## MADRAS

### Compost Scheme

A 2-YEAR SCHEME FOR PRODUCTION OF 88,000 tons of compost in rural areas of Madras at a cost of Rs. 99,000 has been announced by the Director of Agriculture.

The peasants themselves will excavate the pits required and a subsidy of Re. 1 per ton would be paid to them. The ripe compost will be made available to the peasant on his own land at a uniform rate of Rs. 2 per ton. Plans for efficient transport of compost are also being drawn up.

The scheme which is to be worked in all the districts except South Kanara and Nilgiris (where

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# INDIAN PATENTS

The following is a list of a few of the Patent Applications accepted in the *Gazette of India*, Part II, Section 1, for August 1949. Patents from the *Council of Scientific & Industrial Research* are indicated by an asterisk\*.

## Organic Chemicals

- \*39426. DEY, MALLER, PAI & UDUPA: An improved method for the production of 2:4-diaminophenol: *Reduction of m-nitraniline in sulphuric acid at a copper cathode in the presence of copper sulphate.*
- \*39427. DEY, MALLER & PAI: An improved method for the production of 2:4-diaminophenol: *Electrolysing 2:4-dinitrophenol in 30 per cent sulphuric acid at copper cathode.*
- \*39428. DEY, MALLER & PAI: An improved method for the production of 4-amino meta-cresol: *Electrolytic reduction of ortho-nitro-toluene in dilute sulphuric acid in presence of amalgamated monel cathode and catalyst.*
- \*39429. DEY, MALLER & PAI: An improved method for the production of 4-amino-ortho-cresol: *Electrolytic reduction of meta-nitro-toluene in dilute sulphuric acid in the presence of amalgamated monel cathode and catalyst.*
- \*39441. DAMODARAN, RANGACHARI & RAMAKRISHNAN: An enzymic method for the production of calcium gluconate: *Biologically oxidizing glucose in the presence of calcium carbonate by means of glucose oxidase obtained from Aspergillus niger.*
- \*39442. DAMODARAN & SUBRAMANYAN: An improved method for the manufacture of sorbose from sorbitol: *Bacterially oxidizing sorbitol with acetic bacteria in which the medium contains extract of bran or of germinated seeds.*
- \*40261. DEY, MALLER & PAI: Production of 6.6'-dichloro-3'diethoxy-benzidine (Dichloro-o-diphenetidine): *Electrolytically reducing 2-nitro-4-chloro phenetole in aqueous caustic soda removing the hydrazo compound formed by a solvent and treating the hydrazo compound with an acid.*

## Instruments (Professional, Scientific & Controlling)

- 40169. THAPAR & Co. LTD.: Mechanism for manufacture of sheets, block or like built up mica products: *Sprinkling upon conveyor belt thin-rifted mica flakes, supplying adhesive solution to conveyor, when the product of desired thickness is made, it is cut into lengths.*
- 40355. KSHIRSAGAR: Improved bidi-making machine: *Comprises a leaf feeder, a carrier guided by a lever, a set of rollers fitted to a toothed wheel.*

## Metal & Metal Products

- 40803. STANDARD OIL DEV. Co.: Method of treating iron ores: *Ore in subdivided form is brought to a high temperature and melted in dilute suspension in hot gases.*

## Paper & Allied Products

- 39172. ISOLERINGSAKTIEBOLAGET WMB: Method and apparatus for the manufacture of impregnated paper of heat-insulation of multi-layer type: *The paper sheets are dipped in asphalt or bitumen dissolved in a volatile solvent, which is expelled by a current of hot air.*

## Printing, Publishing & Allied Industries

- \*40257. S. SIDDIQUI, J. S. AGGARWAL & MATHUR: Improvements in or relating to manufacture of inks for stamping, printing, stencilling or like purposes: *Resinol or its derivatives, used as sterilizer.*

## Rubber & Rubber Products

- 41453. DUNLOP RUBBER LTD.: Apparatus for the vulcanization of rubber articles in transit: *A chamber connected to two inlet and outlet tubular passages, maintained at a constant vulcanizing temperature through which articles pass over an endless conveyor.*
- 39472. I.C.I. LTD.: Rubber Antioxidants: *Comprising a mixture of bis-(2-hydroxy 3:5-dimethyl-phenyl) alkyl methanes and corresponding bis-(4-hydroxy-2:5-dimethyl-phenyl) alkyl methanes.*
- \*39443. UMA SHANKAR: Creaming of rubber latex: *Hot aqueous extract of tamarind seed powder is mixed with latex and allowed to stand until rubber creams up.*

## Textile & Textile Products

- 41071. THE BRADFORD DYERS' ASSOC. LTD.: Process for reducing the feltability of wool: *Progressive passing of the material through a chamber, continuously feeding halogen gas into the chamber and continuously withdrawing water vapour therefrom.*

## Miscellaneous

- 40816. CARTER: Separation and extraction of stalk from dry tea: *comprises a pair of parallel co-operating toothed rollers with means for rotating the rollers in opposite directions at different speeds.*
- 40817. CARTER: Separation and extraction of stalks from vegetable tissue such as dry tea: *Comprises a tabular casing with a roller having one or more helical V-shaped peripheral grooves rotating with clearance within the casing.*
- 40671. ROY: An improved process for manufacture of pencils: *Casing for the lead rod prepared out of a pliable mass composed of saw-dust or the like made into a dough-like mass by mixing with a binding agent and fed into moulds.*

## Science & Practice of Food Production

MUCH has been written on India's food problem and the necessity and the urgency for attaining self-sufficiency. The problem has been before the people in all its poignancy since the fateful Bengal Famine of 1943. Commissions and committees have examined the many aspects of the problem and recommendations have been made. Fertilizer factories and irrigation projects have been planned and are being speedily executed. The partition of the country has emphasized the need for more intensive effort to step up food production. The crippling effect of importing large quantities of foods on India's national economy has been repeatedly emphasized, and the decision of Government to stop imports from 1951, and the more recent suggestion following the devaluation of the rupee, to stop imports even from 1950, has accelerated Government and private effort to raise production and attain the target of self-sufficiency at the earliest possible date.

Farming and food production are of vital importance ranking with the supply of drinking water and fresh air as an essential for the well-being and efficiency of the nation. The problem of food production concerns all,—the primary producer, the factory worker and the scientist included. In this drive for more and better food, what is the part that science and the scientist have to play? Dispassionate inquiry leads to the conclusion that our resources are adequate to provide sustenance to all, if and only if, the knowledge revealed by scientific research and inquiry is adequately utilized for their planned development.

Science alone cannot bring about increased food supplies, but it can, and must, play a prominent rôle in the achievement of the result. The scientist has the responsibility—nay, the obligation, to develop methods and techniques necessary for the attainment of the objective, viz. production of food of

the quality and in the quantity required to meet the needs of a continuously growing population on a sustained basis and at a price level which can be reached by all. The methods and techniques should be such that they can be adopted and applied effectively within the existing social and economic structure of the primary producer.

This obligation appears to be a formidable one, but the knowledge which inspires the scientist and the faith that sustains him in his endeavours, encourages him to undertake it with confidence. Recent achievements in the United States of America and elsewhere provide the assurance that similar results are attainable in India by harnessing science to farm production.

In any objective examination of the agricultural situation, one thing stands out—agricultural practice lags far behind agricultural science. The most urgent and compelling problem before the scientist is to shorten this lag, to bridge the gap between science and practice, knowledge and application. In an earlier number of the *Journal* we drew attention to the following excerpt from the address by Prof. N. V. Joshi to the Indian Science Congress (1946):

“While the crop yields from improved seeds and milk yields of improved cattle breeds on experimental farms are high, the general level of agricultural and milk yields does not appear to have increased. In what way, then, have we benefited the agriculturist or the agricultural business during all this period of fifty years, from 1890-1940, a period during which research workers in agricultural sciences have accomplished remarkable results which are held by scientists to be of great practical utility? Since we have not benefited the cultivators, it may be concluded that somehow we have failed to convey the information about the results of agricultural research and improvements to the proper quarters or to

get them into common practice by the cultivators."\*

Here is the crux of the problem,— to devise the means to overcome our past failure to convey to the primary producer the methods and techniques of agricultural science in a manner that he can adopt on the field within the means available to him. To say that the farmer is conservative, that he is illiterate and ignorant, is to evade the issue. The fact that he has enthusiastically adopted practices which have produced more and better sugarcane gives the lie to the charge. The failure of scientists to transform the results of soil and crop science to workable farm schedules must be accepted, and the experimental station, which is the scientist's answer for proving and demonstrating the results of research, has obviously proved ineffective.

Discussing the causes that have led to the lag in the use on farms of the best practices of soil and crop management, Dr. McCall of the Bureau of Plant Industry, U.S. Dept. of Agriculture, states :

"When a new product or process is developed in commercial industry, it is axiomatic that an oftentimes extended and expensive period of pilot plant study is necessary before the final economically workable process or equipment is developed from original principles and procedures. This is true no matter how sound and workable the new development may be.

"In the field of agriculture the research scientist has thrown the entire responsibility and expense for the pilot plant stage upon the individual farmer. The farmer is given only the original patent, as it were, and he must adjust the new principles to his own farm and operations. It is true that he may see a preliminary working model which demonstrates the principles on experiment station plots, but this may have little relation to his present farm plan and economic set up. It is up to him to work out the new operation schedules and how he will convert to them. This may require a more or less complete revision of his farm set up. It may mean a change in constituent enterprises in the farm plan and the necessity for him to learn all the basic requirements of the new enterprises with an attendant probability of loss during the learning period. . . . A farmer faced with such a situation is naturally conservative about change except as it fits rather

easily into his present set up. In most cases he cannot be otherwise."\*

The old demonstration farm idea has failed to bring forth the desired results even in a progressive country like the United States. Certain modifications of the idea, however, have been tried and offer possibilities for implementing research. Dr. McCall mentions the following :

Test Demonstration Farms of the Tennessee Valley Authority — A farmer in each community chosen by his neighbours enters into an agreement to carry out certain cropping and fertilizer practices, keeping records which are available to the co-operating State College and Agricultural Experiment Station, the T.V.A., and his neighbours, and permitting his farm to serve as a local demonstration.

State Pilot Farms on which soil management systems indicated by soil research as probably feasible, could be evaluated in economic terms on different soil types and for type of farming factors. As a next step in this plan, community farms similar to the Test Demonstration Farms of the T.V.A. plan would carry the proven findings of the Pilot Farms direct to individual communities.

Individual Farm Advisory Service which would provide in a county or district for well-trained technicians to work with each farmer desiring the service in developing a well-rounded farm plan for his farm. Such a plan to be adequate should cover field layout and farm fences, crops (including varieties), crop rotations, land drainage, liming, fertilizers, tillage schedules for preparing soil in cropping and for weed control and water conservation, erosion control practices, manure conservation and use, timing of all operations, choice and effective use of equipment, numbers and kinds of livestock, animal breeding, feeding and health, preparing and storing feed and other crop materials, housing livestock, facilities for farm processing, crop insect and disease control, and any other pertinent factor.

No one plan, however well conceived, meets all situations. Likewise, no plan however effective in one country proves equally effective when transplanted into another country. It is necessary first to analyse the current practices and examine in what respects they

\* *J. Sci. & Ind. Res.*, 1946, 4, 451.

\* *Freedom from Want*, a symposium, edited by E. E. DeTurk, *Chronica Botanica*, 1948, 11, 276.



have proved defective as instruments of education and research development. It is also necessary to accept the truth in the farmers' complaint that research workers, generally, are out of touch with farming needs and conditions, that the results of research lie published in learned periodicals and bulky tomes and are described in a jargon unintelligible to him, that such publications deal with fragments of the problem and offer no overall solution to his difficulties in practical farming. It should then be possible to work out a plan which would prove operative. In the selection of problems for investigation, or for demonstration on experimental farms, the farmer has so far stood apart from the investigator. It is not always realized that interest which the farmer takes in the development plan is the desideratum for the practical utilization of research. From *prima facie* considerations, a practical approach appears to be one in which the farmer is one of the investigating team, and the farm the experiment station. Research problems should flow from the field to the laboratory and not vice versa.

The raising of abundant crops — either by intensive farming, or by the use of improved seeds, or by any other means — involves the taking away from the soil of large quantities

of nutrients which have to be put back into the soil by a proper system of manuring. This and other aspects of farm management for securing food, and the utilization of our basic capital, namely the soil, so that its productivity is sustained to perpetuity, assumes a certain level of education which the practical farmer should find no difficulty in acquiring if his interest, importance and responsibility as the producer of the nation's vital requirement is adequately recognized. The schemes of craft-centred basic and adult education, which are now being rapidly implemented on a nationwide basis, would be of immense assistance in educating the farmer to adapt himself to the new and the better farming practices.

Sufficient knowledge has accumulated for producing more and better food; what is necessary *now* is to apply it effectively and produce results. In the words of the Hon'ble Minister for Food and Agriculture, if the thousands of public servants manning the agricultural services of our provinces and states function as true guides, philosophers and friends of those who produce the nation's food, the task of making India self-sufficient with respect to the food needs of her rapidly growing population can be satisfactorily accomplished.

## United Nations Scientific Conference on the Conservation & Utilization of Resources (UNSCCURE)

**L**AKE Success, the venue of discussions on international problems, attracted the attention of the scientific world when, on August 17 this year, for the first time in history, 640 scientists, engineers, economists and other experts from 50 countries assembled for a 3-week Conference on the Conservation and Utilization of Natural Resources.

The story of the Conference dates back to the early years of the present century, when the then U.S. President, Theodore Roosevelt, called to conference the Governors of 48 States of America to consider the problems relating to the preservation, protection and

wise use of natural resources of the nation. Several efforts have since been made to convene a world conference of scientists to discuss ways and means of solving problems facing the peoples of our "plundered planet", where resources have been especially drained through wars among nations. Early last year the President of United States put up a suggestion to the U.N. Economic and Social Council to call a United Nations Scientific Conference on Resource Conservation and Utilization. Accepting the suggestion, the Economic and Social Council requested the U.N. Secretary-General on Feb. 11, 1948, to proceed with the plan of the Conference,



"keeping in mind that the task of the Conference is to be limited to an exchange of experience in the technique of conservation and utilization of resources".

During the Conference, 18 plenary meetings and 54 sectional meetings were held. Over 500 technical papers, based on practical experience in the planned use of resources, covering a wide range of fields, — from agriculture to wild-life protection — were presented and discussed.

The following brief résumé of the more important papers presented and discussions held under each section has a special reference to the contribution made by the Indian delegation to the Conference :

Dr. S. S. Bhatnagar, leader of the Indian delegation, who presided over the first meeting of the session, pointed out that "the convening of the Conference was perhaps the greatest work the U.N.O. had done from the scientific point of view". Mr. H. L. Keenleyside, the Canadian Deputy Minister of Mines and Resources, advocated vigorous scientific research on a worldwide basis to tackle problems arising from the possibility of critical mineral shortages in the world in the not too distant future.

The problems facing food production received serious attention. It was pointed out by Sir Herbert Broadley, Deputy Director-General, F.A.O., that to meet the food requirements of the world population in 1960, milk production should be increased by 100 per cent, fruits and vegetables by 163 per cent, meat by 46 per cent, and fats by 34 per cent. He called upon the nations of the world to establish a World Food Fund to "provide resources for accurately measuring the possibilities, organizing the necessary research, planning the strategy of international food campaign and training those upon whom will fall the responsibility of directing the technical operations of the campaign". He further emphasized that the thin layer of top-soil enveloping our planet, which he likened to a tissue paper, "on which depends the fertility of the land and which took from 300 to 1000 years to form", should be carefully preserved and husbanded.

In the subsequent plenary meetings, the delegates examined the application of conservation techniques to mineral, water and wild-life resources; fuel and energy; food and forest products and other resources, with special reference to techniques applicable to under-developed countries of the

world. The sectional meetings devoted particular attention to techniques which would ensure sustained production and enhance supplies by bringing into use resources which were hitherto economically unusable.

*Soil & Forest Resources* — Mr. Hugh H. Bennett, Chief of U.S. Conservation Service, pointed out that the productive capacity of good land, which was limited to 4,000,000,000 acres, was being reduced by abusive use in many countries. He emphasized the urgency of classifying land according to its best use.

Dr. J. N. Mukherji, Director, Indian Agricultural Research Institute, New Delhi, speaking on soil productivity in tropical climates, stated that a rotation crop system suited to the individual soil type was no less important than the use of fertilizers. In the absence of adequate supplies of inorganic fertilizers, green manures are specially important for ensuring soil productivity.

Reclamation and effective use of new agricultural lands present several problems for solution. Dr. R. Sethi, Agricultural Development Commissioner to the Government of India, emphasized that attention to malaria control is essential for the development of new lands. He stressed also the need for securing suitable machinery for the reclamation of land in India.

The provision of improved tools and implements for small-scale farming would prove effective in augmenting food supplies. A paper by Mr. Mason Vaughn, presented before the Conference, described a soil-inverting plough and a small cultivator with suitable attachments for both, which would meet practically all the needs of the small farmer.

The part played by forests in the protection of resources was discussed at one of the sessions of the Conference. Mr. C. R. Ranganathan of the Indian Forest Service pointed out that more attention should be paid to hydrological and protective aspects of forests than hitherto and expressed the view that State ownership was essential for securing the best results.

*Preservation and chemical utilization of wood resources* formed the subject of a paper by Mr. J. F. Harkam of the Department of Mines and Resources of Canada. Preservative treatment increased the life of timber in service 3 to 4 times, reduced drain on forests, increased the utility of wood, and opened up a market for timber products derived from species of low natural durability.

**Fisheries** — The delegates exchanged views on pond culture, i.e. the systematic raising of fish farms, which can, under favourable conditions, be an important source of food and a lucrative industry.

Dr. S. L. Hora, Director of the Zoological Survey of India, presented a general review of the problem. Describing the multiple uses of natural ponds, Dr. Hora stated that in Philippines alone there were 600,000 hectares of undeveloped fresh and salt water swamps capable of producing 200 million fish annually. He estimated that even if only 10 per cent of the money, material and men now employed in considering the ways and means of developing marine fisheries in the tropics were diverted to improve ponds for fish culture, the result would go a long way in solving the food problem of the world.

In this connection, Dr. Hora drew attention to the cultivation of fish in rice-fields as practised in Japan, which has benefited paddy crops by as much as 10 per cent. By proper embankments, vast low-lying areas in the vicinity of sea coasts could be made productive. Besides their use for raising fish crops, the areas could be converted into salt ponds which, when silted up, would become fresh-water ponds and finally rice-fields.

Coming to the conservation of marine resources, Dr. H. Srinivasa Rao of the Central Marine Fisheries Research Station, Madras, pointed out that the integration of hydrological, biological and related studies is a feature of the fisheries research programme in India. Exploitation, rather than conservation, is the immediate problem of India's marine fisheries. Nevertheless, the Government is taking a long-range view of the problem and taking steps to obtain scientific data necessary for development programmes which would lead to sustained yields from this resource.

**Minerals & Metals** — Geologists were unanimous in the opinion that the world's store of metals would continue to contribute to better living conditions only if the "shocking" waste caused by war was done away with, and if national and political boundaries, which stand in the way of international co-operation, were removed.

In a paper entitled *Metals in Relation to Living Standards* Dr. D. N. Wadia revealed that more basic metals have been used up since 1914 than during the whole of human history.

During a discussion on metal reserves of the United States, it was estimated that the commercial reserves of iron in the United States would be consumed within 75 years and of other important minerals and metals possibly in 1 to 19 years.

The undiscovered reserves of petroleum have been estimated to be 150,000 million barrels, i.e. about 500 times the current annual consumption, but this estimate has been considered to be too high. Mr. Aguilar, the Mexican oil expert, urged the need for setting up of an international committee under the United Nations or any other agency to undertake a world survey of oil reserves.

The topics discussed at the sectional meetings on *Fuels and Energy* included the use of giant turbines for harnessing wind power to generate electricity; methods of overcoming critical shortages of coal; and techniques used in the United States, France and England to stretch their coking coal reserves.

Problems connected with the development of river basins; importance of forests and plant cover in controlling floods; river navigation as a means of transport in underdeveloped countries; and irrigation and drainage in relation to food production were among the other problems that engaged the attention of the delegates in the various sections of the Conference.

The example of the Tennessee Valley Authority in the development of a river basin formed the subject-matter of a pamphlet presented at the Conference.

**New Resources** — The possibilities of tapping new resources to meet the needs of industry were examined in some detail during the Conference. Dr. Neville Woodward, Director of the Scottish Seaweed Research Institute, drew attention to the fast-developing fields of *Chemurgy* which deals with the utilization of agricultural products and wastes as raw materials for secondary industries. Recent developments in seaweed utilization were described.

**Appraising Resources** — Resource appraisal requires the application of systematic inventory procedures for which surveyors had to collaborate more closely with official agencies responsible for development projects. Dr. P. C. Mahalanobis of the Indian Statistical Institute, dealt with the application of statistics to resource appraisal and utilization.

**Resources Conservation in Under-developed Countries** — Dr. Hernan Santa Cruz of Chile

led the discussion on the problem of techniques for conserving resources in the less developed countries. Measures adopted for conservation of resources in these regions were inadequate, and foreign capital has possibly accelerated the "intensity" of exploitation of resources. The world prices of commodities were fixed without consulting the producing countries with the result that the prices realized were not such as to raise the standard of living of the producers.

Dr. S. S. Bhatnagar sounded a note of caution against transplantation, without precaution of techniques devised for, and specially suited to, a highly industrialized country to another where manpower was cheap and plentiful. He stressed that the less developed regions would be benefited if attention is paid to the following:

- (1) Training and acquisition of technical personnel.
- (2) Procurement of literature, equipment and machinery.
- (3) Means to make foreign and indigenous capital flow into less developed countries without the imposition of conditions which might be considered humiliating by nations requesting help.

(4) Application of technical knowledge already in existence in order to improve the process techniques which are indigenous; and

(5) progressive measures in the legal and political fields, without which it was possible to "stifle" any technical or scientific activity.

Of special interest among other papers discussed at the Conference were those dealing with the production of food yeast and artificial foodstuffs. Another paper described recent developments in air surveys and aerial photography which made it possible to carry out rapid surveys of forest resources.

The success of the Conference, as Dr. Bhatnagar stated, will be determined by "the rate at which the less developed regions 'catch up' with the developed regions". In the words of the Secretary-General, one of the most important results of the Conference had been "to demonstrate to the peoples of the world that the United Nations Organization was concerned with building the economic and technological foundations of international peace by enlisting the collaboration of the scientific and technical world".

## Characteristics of the Ionosphere over Calcutta (August 1949)

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(MRS.) T. K. BHAR & A. P. MITRA

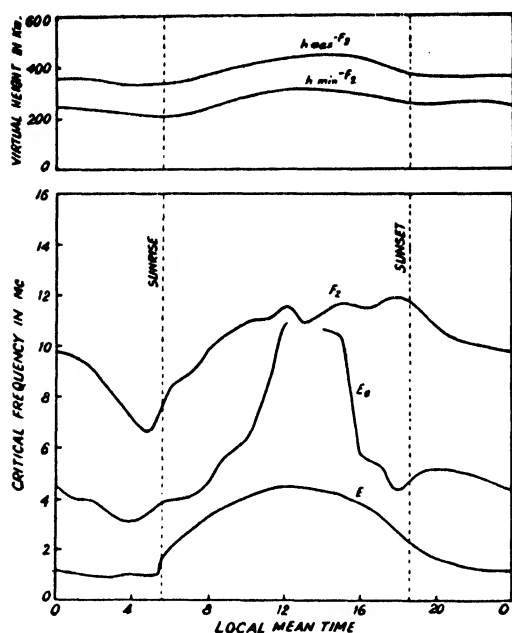
*Wireless Laboratory, University College of Science, Calcutta*

**T**HE following are the ionospheric data observed at Calcutta for the month of August 1949.

Fig. 1 represents the mean hourly values of the penetration frequencies of the E and E<sub>s</sub> regions and the penetration frequencies and virtual heights of region F<sub>2</sub>. The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the prediction of the maximum usable frequencies which can be used for different distances of transmission, by reflection at the F region

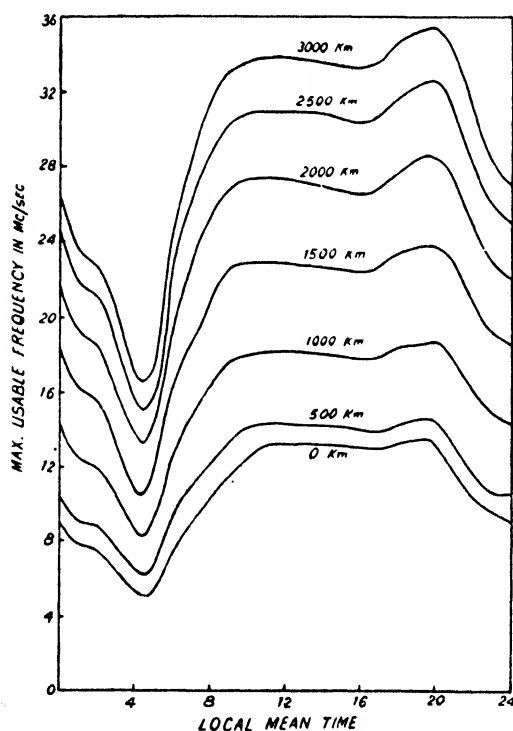
over Calcutta for the month of November 1949. Table I gives the different occasions during routine observation when sporadic E ionization was observed and the values of the corresponding penetration frequencies and heights.

Sporadic E ionization was found to occur frequently during early morning and also in the afternoon hours. Absorption of normal E echoes at mid-day was less than in the previous month. The behaviour of region F<sub>2</sub> was found to be normal in this month except for the last few days.



( 5 HOURS 54 MINUTES AHEAD OF G.M.T. )

FIG. 1 — AUGUST 1949.



AT POINT OF REFLECTION.

FIG. 2 — PREDICTED M.U.F. VIA  $F_2$  LAYER, NOVEMBER 1949.

TABLE I

MONTH & YEAR	DATE	HOUR	$f_o E_s$ Mc.	$h E_s$ Km.
August 1949	1	08.00	4.75	120
		09.00	6.10	120
		10.00	6.80	135
		11.00	7.10	135
		18.00	5.50	135
		20.00	6.00	120
	2	06.00	4.50	105
		08.00	4.50	120
		09.00	6.75	120
		10.00	7.00	120
		16.00	6.60	120
		17.00	4.75	120
		18.00	3.60	120
		19.00	4.25	105
		20.00	4.00	105
	3	08.00	4.55	120
		09.00	6.00	120
		10.00	4.50	120
		11.00	6.25	135
	4	11.00	6.80	135
		16.00	6.80	135
		18.00	4.50	120
		23.00	2.85	105
	5	00.00	3.25	90
		04.00	3.00	90
		05.00	3.65	90
		19.00	3.00	105
	6	07.00	4.50	120
		09.00	6.10	120
	8	09.00	4.20	120
		12.00	11.00	150
		16.00	6.00	135
		17.00	7.10	135
		18.00	6.00	120
		19.00	6.25	120
		21.00	6.00	120
		22.00	5.25	105
		23.00	5.35	105
	9	00.00	8.15	105
		01.00	5.20	105
		02.00	4.50	105
		03.00	3.25	90
		07.00	4.25	105
		08.00	5.25	120
		09.00	6.00	120
		10.00	8.00	120
	9	11.00	9.75	135
		13.00	10.70	150
		14.00	10.70	150
		15.00	10.50	150
		16.00	10.00	150
		20.00	10.00	150
		21.00	5.25	135
		22.00	8.00	135
		23.00	6.25	135
	10	00.00	4.00	120
		07.00	3.00	120
		08.00	5.00	120
		10.00	6.00	135
		13.00	12.40	150
		16.00	5.75	150
		17.00	5.20	135
		18.00	4.70	135
		19.00	3.50	120
		22.00	2.70	105
	11	08.00	4.20	90
		09.00	5.00	120
		10.00	5.40	120
	18	13.00	10.95	150
		16.00	3.35	135
		17.00	3.20	120
		18.00	3.20	105
	19	09.00	4.70	120
		17.00	5.85	120
		21.00	6.20	120
		22.00	5.70	105
		23.00	4.20	105

TABLE I—contd.

MONTH & YEAR	DATE	HOUR	$f^\circ E_s$	$h^\circ E_s$
			Mc.	Km.
August 1949	20	09.00	6.70	120
	22	11.00	10.40	150
		20.00	6.25	135
		21.00	5.00	120
	23	18.00	3.40	120
		23.00	3.30	90
	24	00.00	3.40	90
		01.00	3.50	90
		02.00	3.30	90
		05.00	3.80	90
		06.00	4.70	105
	25	17.00	6.55	120
		18.00	3.40	120

TABLE I—contd.

MONTH & YEAR	DATE	HOUR	$f^\circ E_s$	$h^\circ E_s$
			Mc.	Km.
August 1949	25	19.00	3.50	120
		20.00	4.25	120
		21.00	4.50	105
		23.00	4.75	105
	26	00.00	4.00	105
		01.00	3.20	90
		20.00	3.25	105
		21.00	3.20	105
	29	20.00	4.00	105
		22.00	4.25	105
	30	07.00	3.75	120
		18.00	4.80	120
		23.00	4.00	90

## Design of All-India Community Radio Receivers for Villages

H. JOGA RAO & K. SRINIVASAN

*Provincial Broadcasting Department, Government of Madras, Madras*

THE Provincial Broadcasting Department of the Government of Madras started installation and maintenance of community radio receivers in villages in 1938. About 100 sets were installed in 1938-39, as an experimental measure, after the installation of 5 kW. medium-wave transmitter at Tiruchi and 10 kW. short-wave transmitter at Madras.

Subsequently, Government of Madras sanctioned a scheme for installing 300 sets in the villages. The maintenance of these sets is the responsibility of the Provincial Broadcasting Department. The community sets installed in the province increased year by year and, by 1947, there were about 700 sets installed.

Early 1946, the Government of India announced a basic plan for providing a large number of additional transmitters throughout India. The Government of Madras had also contemplated installation of the community receivers on a large scale in various villages in the province to enable the villages to avail of these increased broadcasting facilities. Early in 1947, a comprehensive scheme was, therefore, drawn up

by this Department for installing 8,000 sets in as many villages.

### Problems of Large-scale Installation of Community Radio Receivers

The important aspects of rural broadcasting are: (i) keeping down the initial cost of installation of a community set; (ii) lowering the recurring cost of maintenance of the set; (iii) policy as regards: (a) the replacement of the sets at the end of their useful life, (b) effecting a change from a battery-operated set to a mains-operated receiver whenever a village is electrified, at low cost; and (iv) the minimum requirements of reception with respect to wavelengths and output which a community receiver should fulfil.

(i) *Initial Cost of Community Set*—The Provincial Broadcasting Department has been purchasing, as and when necessary, various types of commercial, domestic, 5 or 6 valves, battery or mains-operated radio receivers of American or British manufacture for installation in the villages. Prior to 1941, the cost of such a receiver was ranging between Rs. 150 to Rs. 250 and

from 1941 to 1944 from Rs. 300 to Rs. 350. In 1944 about 600 lease-lend receivers (for which only "out of pocket expenses" were paid) were obtained by the Madras Government from the Government of India at cost varying from Rs. 88 to Rs. 142, and they were installed in various villages.

Extra cost is incurred by the Department before the installation of the sets in the villages for carrying out certain modifications. They are mainly as follows :

1. An insect-proof frame with a lock, for the back of the set, to protect it from insects and to avoid tampering of the internal mechanism.

2. The community sets are designed to receive programmes of either Madras or Tiruchi stations depending upon the language spoken by the local people. The tuning of the sets, therefore, was adjusted to either of these stations, so that their operation is simplified.

3. An additional cabinet or horn-type baffle for the loud speaker of the set, with an extension cable 15' in length, is provided, so that even though the set is installed in a room, the loud speaker can be brought out to the open to serve the listeners in the open. These modifications limit the working of the sets to the essential purposes of community listening.

(ii) *Cost of Maintenance* — As the number of sets installed in the province rose from about 100 in 1940 to about 700 in 1947, the cost of maintenance per set per year assumed importance. Apart from the expenditure on common items like transport, establishment, etc., items such as (1) cost of spares used for servicing ; (2) types and design of sets (whether simple, or complicated, using exclusive types of components, etc.) with respect to their simplicity for servicing and maintenance assumed greater importance. Table I gives the types installed and maintained by this Department in 1948.

The large variety of sets to be maintained compelled the Department to keep stock of a variety of spare parts, e.g. valves, band switches, condensers, etc., some of these being special to a given model of the receiver.

Another important factor contributing to the cost of maintenance is the non-essential electrical and mechanical parts in the commercial types of sets, which are superfluous in a community set. The commercial sets have highly finished and costly cabinets, multi-band tuning and dial-drive mechanisms, band spread circuits, tuning ranges covering

TABLE I — RADIO RECEIVER MODELS

BATTERY MODELS				MAINS MODELS			
No.	Manufacturers	Model		No.	Manufacturers	Model	
1	R.C.A.	8QB		1	R.C.A.	8Q2	
2	R.C.A.	QB1		2	R.C.A.	8Q1	
3	R.C.A.	7QB		3	R.C.A.	85T8	
4	R.C.A.	85BT		4	R.C.A.	9Q4	
5	R.C.A.	QB9		5	R.C.A.	Q11	
6	Philco	38-624		6	Philco	38-620	
7	Philco	40-748T		7	Philco	37-2670	
8	Philco	39-744		8	Philco	40-780T	
9	G.E.	1B-72		9	G.E.	HE 74	
10	G.E.	G1E 63		10	G.E.	40-11E 740	
11	H.M.V.	699		11	G.E.	X 115 C.A.	
12	Marconi	822		12	Cossor	FX 396	
13	Andrea	E6B		13	H.M.V.	673	
14	Pye	V41RF		14	H.M.V.	675	
15	Lincoln	Mains and battery		15	H.M.V.	5211	
16	Crystalltone	7906		16	Marconi	829	
17	Philips	739 VN		17	Roburn		
18	Philips	663 VN		18	Philips	HN 436	
				19	G.E.C.	BC 4172	
				20	Westing house	M116A	

wide frequency bands, and other gadgets like push-button tuning, etc., which, even though not necessary for a community set, have to be maintained to keep the set as a whole in good condition. As the designs of commercial sets are complicated, and a large number of different types of sets had to be maintained, skilled technical staff became necessary, and the number of sets under the charge of any one person had to be restricted to about 20. The commercial sets are not designed for frequent transport over long distances by road or rail, whereas the sets installed in the interior villages have had to be moved from place to place and they frequently developed faults due to exposure or jolting.

(iii) *Cost of Replacements* — Another important aspect of rural broadcasting is that some of the sets installed in 1938 have completed nearly 10 years of service. Some of the sets had to be condemned as unfit for use for the following reasons :

1. Important original components like tuning coils, band switches and other circuit components were no longer available for servicing the sets nor were standard components available in the market.

2. With the progress of rural electrification, a certain number of battery sets will have to be replaced by mains-operated receivers every year.

(iv) *Basic Requirements of a Community Receiver* — Taking into consideration the various aspects of rural broadcasting, it was considered imperative to evolve a standard type of community set, if installation and maintenance of community receivers in thousands of villages have to be successfully and economically carried out. The requirements are :

1. The initial cost of installation should be low.
2. The maintenance cost of the sets should be low.
3. The set should have a long life. It should not be out-moded with time for want for any particular spare part.
4. The cost of conversion from battery to mains-operation should be low.

To meet these requirements, the design of a suitable receiver was taken up early in 1947 in the Research Section of the Provincial Broadcasting Department.

#### Standard Type of Community Radio Receivers

A superheterodyne type was decided upon in preference to a straight receiver owing to the greater sensitivity, selectivity and stability of the former. The provision of reaction control for increasing the sensitivity of a straight receiver may result in instability in the set, thus causing disturbance through radiation.

**Stages in the Set** — Keeping in view the economy and simplicity in construction, the following essential stages are provided without sacrificing the sensitivity and selectivity : (i) first detector and oscillator ; (ii) intermediate frequency amplifier ; (iii) second detector and audio voltage amplifier ; (iv) audio driver amplifier ; and (v) power output.

Power pack is built as a separate unit. A complete circuit diagram and the values of components are given in Fig. 1 and Table II.

The components used are standard types readily available in the market.

**Controls** — The number of controls necessary to operate the various stages has been reduced to a minimum in order to simplify the handling of the set by a village operator. A heavy-duty main switch, separate from the volume control, is provided so that it may not be easily spoilt by frequent use and to handle the heavy current that flows through when the set is working off a 6-volt battery. The usual type of continuously variable volume control is provided. No tone control is required as the set is designed for balanced tone output which meets the requirements of community listening in public parks, etc. An air dielectric low-capacity (0.50 p.f.) tuning condenser (incorporated in the oscillator grid circuit) is also provided. These controls, viz. main switch, volume control and tuning control are fitted to a panel in the front of the chassis for easy manipulation.

**Power Pack & Audio Power Output Rating** — In view of the necessity for providing an easy change-over from battery to mains operation when required, the power pack is built separately and fitted to the chassis frame so that it can be taken out without disturbing the wiring or the layout of the main receiver circuits. The power pack is connected to the receiver circuit by means of a non-reversible plug and socket and a short length of screened multi-core cable. The socket in the receiver chassis is common and the plug

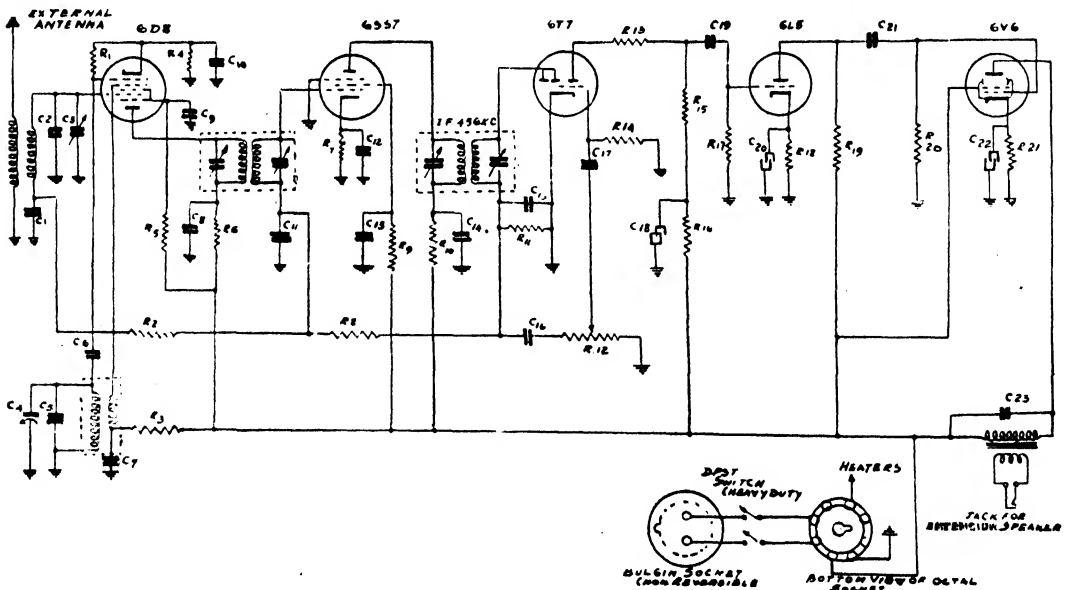


FIG. 1 — CIRCUIT DIAGRAM OF COMMUNITY RECEIVER.

TABLE II—COMPONENTS &amp; THEIR VALUES

Receiver Section	
R. 1 35 k. ohms	C. 1 0.04 $\mu$ F paper
R. 2 2 megohms	C. 2 150 p.f. mica
R. 3 20 k. ohms	C. 3 30 p.f. mica trimmer
R. 4 350 ohms	C. 4 30 p.f. air trimmer
R. 5 25 k. ohms	C. 5 150 p.f. mica
R. 6 5 k. ohms	C. 6 100 p.f. mica
R. 7 350 ohms	C. 7 0.01 $\mu$ F paper
R. 8 1 megohm	C. 8 0.01 $\mu$ F paper
R. 9 200 k. ohms	C. 9 0.05 $\mu$ F paper
R.10 20 k. ohms	C.10 0.02 $\mu$ F paper
R.11 500 k. ohms	C.11 0.02 $\mu$ F paper
R.12 1 megohm volume control	C.12 0.03 $\mu$ F paper
R.13 20 k. ohms	C.13 0.01 $\mu$ F paper
R.14 5 megohms	C.14 0.01 $\mu$ F paper
R.15 50 k. ohms	C.15 250 p.f. mica
R.16 100 k. ohms	C.16 0.01 $\mu$ F paper
R.17 200 k. ohms	C.17 0.04 $\mu$ F paper
R.18 2 k. ohms	C.18 8 $\mu$ F, 50 W.V. dry electrolytic
R.19 50 k. ohms	C.19 0.01 $\mu$ F paper
R.20 100 k. ohms	C.20 8 $\mu$ F, 50 W.V. dry electrolytic
R.21 500 ohms	C.21 0.01 $\mu$ F paper
	C.22 8 $\mu$ F, 50 W.V. dry electrolytic
	C.23 0.01 $\mu$ F paper

**Battery Power Pack Section**

T. 1 Triller, Synchronous, 6-volt operation, 6-pin base	
C.24 0.2 $\mu$ F paper	
C.25 0.01 $\mu$ F mica moulded	
C.26 0.05 $\mu$ F paper	
C.27 0.01 $\mu$ F paper	
C.28 40 $\mu$ F, 450, W.V. dry electrolytic	
C.29 40 $\mu$ F, 450, W.V. dry electrolytic	
R.22 2500 ohms	No load primary current 0.35 amp. for ma. secondary current
R.23 200 ohms	
RFC R.F. choke 20 m.H.	at 160 volts primary drawn 1.15 amp.
T. 2 Vibrator transformer 1 35 ratio	

**A.C. Power Pack Section**

T. 3 Power transformer	230-0-230, 50 ma.
R.24 1000 ohms, 5 watts	
C.30 16 $\mu$ F 450 W.V. dry electrolytic	
C.31 16 $\mu$ F 450 W.V. dry electrolytic	

from either mains or battery power pack can be directly plugged into it. As the set has to be worked either off 220-volt main or a 6-volt battery, the valves are so chosen that their heater currents as well as the H.T. current drain is low to keep down the battery current within limit. The working H.T. voltage in the set is stabilized at 160 volts for both mains and battery operation. At the same time, the maximum undistorted power output of the set, while working on battery, does not fall below 1.25 watts. The sensitivity of the set is also maintained in the order of 20  $\mu$ v. for 500 m. watts output.

**Design of the Circuits**

**First Detector Stage** — As the set is mainly required for spot frequency reception, that

is to tune to one station only at a time, it is provided only with a tuning arrangement to cover a range of about 100 kc./s. on either side of the chosen spot frequency.

The aerial input is fed to the tuned grid circuit of the frequency changer valve 6D8. As already explained, this particular valve is selected for its low heater drain of 0.15 amp. The tuned grid circuit is conventional rejector type circuit, the parallel capacity being in this case a mica trimmer condenser (0.30 p.f.) and with another low-loss fixed mica condenser (about 150 p.f.) in parallel. The aerial coil is of high impedance section wound type (1.25 mh). The coil former is made of 1" outside diam., low-loss ebonite tube with  $\frac{1}{8}$ " wall provided with suitable brackets for fixing it to the chassis. The grid circuit is pre-tuned by means of the trimmer condenser for the required signal frequency. The Q factor of this air core coil is of the order of 100 ensuring fair stage gain and sufficient selectivity.

The coil tried was at first of the plug-in type with pin connections at the base and there were 2 separate coils, one for medium and the other for short-wave bands. Later it was found that the plug-in coils develop high resistance contacts at the base pins and can be advantageously replaced by coils mounted on the chassis permanently and connections made through a simple band switch.

**Oscillator Circuit** — The oscillator is of the conventional type with tuned grid circuit and a feed-back coil in the anode. This is an air core coil, wound on a 1" ebonite former as in the case of the aerial coil and it is shielded by a cylindrical copper shield of  $2\frac{1}{4}$ " diam. made of 12 SWG sheet. The parallel capacity in the circuit is provided by means of a fixed mica dielectric condenser in parallel with an air dielectric variable condenser of 5-50 p.f. range. This variable condenser is robust, with an extension shaft for manual tuning. This condenser is specially provided to give a range of 100 kc./s. variation in the oscillator frequency. This enables tuning of the set within a range of 100 kc./s., about the spot frequency for which the aerial coil is tuned. It is seen, therefore, that this control of the oscillator frequency which can be manipulated by the operator enables him to retune the receiver properly, compensating for any tuning drift in the set. At the same time, this device has simplified the tracking problem in the set as the band width is limited to 100 kc./s.



unlike in the case of a broadcast receiver where the band width is about 1,000 kc./s. on broadcast band and 6 to 7 mc./s. on medium short band. On account of this, the gang condenser in this community set design could be eliminated. This simplification in the circuit enables the alignment of the aerial trimmer at any time to be done directly while receiving the signal from the station for which the set is pre-tuned. The oscillator grid condenser, on being tuned for maximum output, automatically tracks the oscillator frequency in relation to the signal frequency. Therefore, the usual multi-band signal generator is not necessary for the alignment of the set while servicing it. A compact oscillator generating the intermediate frequency will only be necessary for the occasional alignment of the I.F. circuits of the set. This simplification in the design has the added advantage of covering two spot frequencies in broadcast band and a short band width of 100 kc./s. in each of 31, 41, 49 and 60 metre bands with the help of only two coils with fixed capacitors connected through a band switch. This enables easier servicing and checking of the circuits when necessary, than in the case of the usual commercial receivers with 5 to 8 bands including spread band circuits, etc. Most of these extra circuits in commercial sets are superfluous for this purpose, and involve complicated servicing work.

**Intermediate Frequency Stages** — The intermediate frequency stage in the set is of the conventional design using 6SS7 (0.15 amp. heater) and the frequency is 456 kc./s. The transformers used are Meissner standard type Nos. 16-5712 and 16-5714. They are air-cored types with ceramic base and mica dielectric trimmers. The  $Q$  factor of the coils in these transformers is between 85 and 110. The circuit impedance of the first I.F. transformer is 0.4 megohm corresponding to an  $r/L$  ratio of 30,000. The primary impedance of second I.F. transformer is also 0.4 megohm corresponding to an  $r/L$  ratio

of 26,000. These figures compare favourably with those that are met with in the usual commercial broadcast receivers (TABLE III).

**Second Detector Stage** — The second detector is 6T7 valve (0.15 amp. heater) in which the two diodes are connected together to one side of the secondary of the second I.F. transformer and circuit being complete to cathode and earth through a load resistance of 500,000 ohms. The audio voltage is fed through a volume control to the grid of the triode section of 6T7. Simple AVC voltage is fed to the grids of the previous I.F. amplifier and frequency changer valves.

The triode audio amplifier of 6T7 is designed with a high grid leak resistance (5 megohms) to reduce the shunting effect on the detector load. The anode of the audio amplifier is sufficiently decoupled to increase the stability of the stages. The voltage gain in the stage is of the order of 35.

**Audio Driver Stage** — A simple triode voltage amplifier stage using 6L5 valve (0.15 amp. heater) is incorporated to give sufficient drive to the output stage. This is considered advantageous in keeping down voltage gain to be obtained in the previous stage, thus reducing the tendency for instability. This stage also enabled the overall sensitivity of the set to be increased without sacrificing stability. The increase in the battery current owing to this stage is comparatively small (0.15 amp. heater current; about 5 ma. plate current) and the total battery current does not exceed the specified amount of 2.9 amp. for the set. The voltage gain of this stage is 12.

**Output Power Stage** — In the output power stage, a beam power output valve 6V6 (0.45 amp. heater) is used for the following consideration. It is worked as a class A power amplifier with cathode resistance for generating the grid bias. This valve is rated at 2 watts with 180 volts anode voltage with a maximum grid voltage swing of 8.5. In order to economize in the H.T. anode current

TABLE III — COMPARATIVE DATA OF MEISSNER & QB9 I.F. TRANSFORMERS

TRANSFORMER	FREQUENCY kc./s.	PRIMARY					SECONDARY			GRID COUPLING FACTOR	EXISTING COUPLING FACTOR	MUTUAL INDUC- TANCE m.H.
		Induc- tance (L) m.H.	Tuning capacity p.f.	Q	r/L	L/cr ohms	Induc- tance (L) m.H.	Tuning capacity p.f.	Q			
QB9 1st I.F.	455	1.05	117	86	33,240	258,000	1.09	111.4	82	0.012	0.019	0.021
Meissner 1st I.F.	456	1.42	84.8	98	30,100	394,000	1.302	86	85	0.011	0.012	0.017
QB9 2nd I.F.	455	1.85	66	85	33,620	449,400	1.95	62.2	82	0.012	0.02	0.0392
Meissner 2nd I.F.	456	1.247	96	112	25,800	403,500	1.174	94	95	0.0026	0.002	0.273

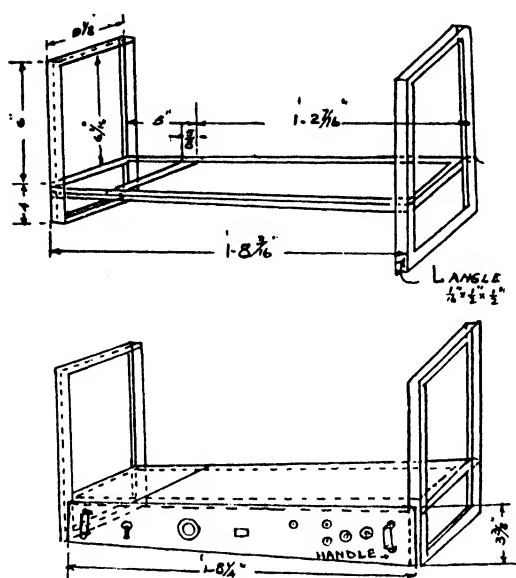


FIG. 2 — CHASSIS FRAME.

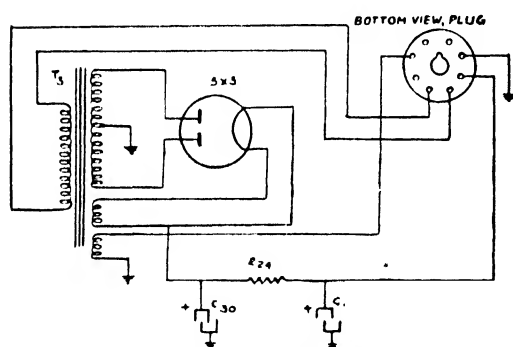


FIG. 3 — MAINS POWER PACK CIRCUIT DIAGRAM.

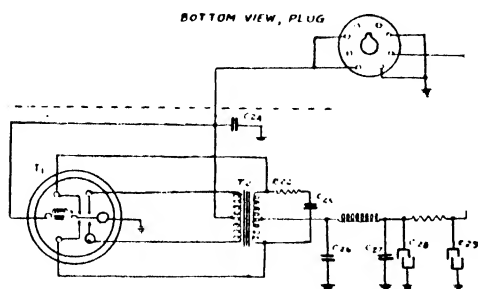


FIG. 4 — BATTERY POWER PACK CIRCUIT DIAGRAM.

of this valve at the same time obtaining not less than 1.2 watts maximum output, the circuit constants in the stage are so

adjusted as to give the following operating conditions :

- (i) Anode voltage, 150
- (ii) Anode current, 25 ma.
- (iii) Cathode voltage, 9
- (iv) AC grid volts ( for 1.2 watts output ), 12 volts.

The question of economy in the cost of the set has also been taken into consideration in deciding upon this beam power class A output stage in preference to a push-pull output stage. A triode or pentode push-pull output stage will require additional components and circuit in the shape of either a driver transformer or a phase inverter circuit. A suitable valve for the push-pull output stage will be 6Y7 driven by a 6W7 with the help of a transformer. It may have an advantage of giving maximum output with less percentage distortion than a class A beam power tetrode used in this design. But on practical testing of the performance of this set, it is seen that the 6V6 class A output stage has given an equal performance as in the case of 6Y7 output stage, at the same time economizing in the cost of production. The test data of the output given by this set and the current drain compared to those of a similar commercial set using 6SA7, 6SS7, 6T7, 6W7 and 6Y7 are given in Table IV.

**Power Pack Assembly** — The power pack is assembled separately so that it can be fitted in the chassis frame of the set adjacent to the main receiver portion as shown in Fig. 2. The overall dimensions of the base plate of the battery and mains power pack are kept the same so that they are interchangeable. In Fig. 3 the circuit of the mains power pack is shown. Each power pack is self-contained with its filter circuits. The main switch fitted in the control panel is

TABLE IV — TEST DATA FOR R.C.A. QB9 SET ;  
BATTERY OPERATED  
( with permanent magnet speaker )

Test frequency — 760 kc./s. Modulated 30% with 400 cycles A.F.

Sensitivity —

Input for 500 m.w. output	56 $\mu$ v.
High tension voltage supply to the bands	105 volts
Maximum output at 760 kc./s.	1.2 watts
L.T. current drain off 6-volt battery	2.2 amp.

Test Data for Community Receiver —

Sensitivity ; Test Frequency	1200 kc./s. modulated 30% with 400 cycles A.F.
Input for 500 milliwatts output	20 $\mu$ v.
High tension voltage supply to the anodes	160 volts
Maximum output at 1200 kc./s.	1.25 watts
L.T. current drain off 6-volt battery	2.1 amp.

wired to the input socket fitted in the receiver chassis. From the circuits of these power packs given in Fig. 1, it may be seen that the mains or battery lead, H.T. lead and valve heater supply lead are brought out to the various pins of an 8-pin plug. The connections to these plugs are made through a short multi-core shielded cable. The connections to the 8-pin socket fitted to the receiver chassis are so wired that the plug of either the mains or battery power packs can be plugged-in direct so that the set is ready for instantaneous operation.

**Chassis & Other Mechanical Parts** — The simplification in the design resulting in the elimination of dial, dial drive gang condenser, etc., enables the use of a frame type of chassis for the whole set. The power pack is mounted in a separate sheet from the main receiver and can be screwed down to the chassis frame, a sketch of which is given in Fig. 2. The battery power pack (Fig. 4) and the mains power pack are thus interchangeable. The shape of the frame-chassis is such that the set can be rested in any position for quick servicing without damaging the valves or other components. It is robust and at the same time light, being built of aluminium L angle. It has been made slightly larger than a conventional chassis of usual 5-valve commercial receiver to provide room for clear and neat wiring and for facilitating quick and easy servicing. The loud speaker is not mounted in the receiver frame but is housed in a separate cabinet for installation away from the set. The loud speaker is provided with a 15' twin core extension cable ending in a 2-pin non-reversible plug, and is plugged into a corresponding socket provided in the control panel of the receiver to connect it to the output of the set. The controls for the set and the aerial-earth terminals are also fitted in the front panel for easy accessibility. The

receiver chassis frame goes into a simple and strong wooden cabinet which is made insect and dust-proof. The receiver cabinet has been constructed with the front door in two halves, so that by raising the lower half, the controls alone are accessible to the operator. The top half is closed and sealed until required to be opened for taking out the set for servicing, only when necessary.

This design of the simple community set meets the following requirements :

1. The general design of the circuits, layout of the components and the mechanical parts are simple such that they can be built in any moderately equipped workshop and laboratory.

2. No special skilled labour or special plant and machinery will be required to produce these sets even in bulk.

3. The design of the set is directly suited to community listening purposes such that no further modifications are necessary.

4. The cost of maintenance will be considerably lower due to simplification in the stocking of spare equipment required for servicing and ease of servicing.

#### Acknowledgements

Our thanks are due to Shri V. V. L. Rao, Permanent Radio Engineer, Provincial Broadcasting Department, now General Manager, *Messrs Radio & Electricals Ltd.*, Madras, who has evinced continued interest in this work. Our grateful acknowledgements are due to the Government of Madras for affording necessary funds and facilities for carrying out this work in the workshop and laboratories of the Provincial Broadcasting Department. Our thanks are due to the members of the staff in charge of office, laboratory and workshop of this Department, who have given every co-operation and help to bring this work to a successful conclusion.

# Manufacture of Sponge Iron— Literature Report No. 2

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B. R. NIJHAWAN & P. K. GUPTE

*National Metallurgical Laboratory (C.S.I.R.), Jamshedpur*

**S**PONGE iron is the product of direct reduction of iron ore below the fusion point of iron. Considerable work has been carried out in the United States (mainly by the *U.S. Bureau of Mines*), Sweden, Germany and France, stress being laid mainly on the following important aspects :

(1) Sponge iron as a substitute for scrap in the production of steel, especially in electric smelting furnace for the manufacture of high-quality steel<sup>1-4</sup>.

(2) Utilization of non-coking type of coal and other gaseous fuels for the reduction of iron ores. This problem is of special interest to India ; she has ample high-grade iron-ore deposits but lacks coking coal.

(3) Use of sponge iron in chemical and metallurgical operations requiring finely divided metallic iron of high porosity. Sponge iron has many advantages as a precipitant. Precipitation of metals from solutions as used in copper metallurgy has been advocated for many years.

## Methods of Manufacture

1. *Methods Developed by the United States Bureau of Mines* — The experimental work of the *Bureau* deals mainly with the production of sponge iron by heating iron ores at low temperature using solid carbon as the reducing agent.

The small-scale experiments are carried out in muffle furnaces. The reduction temperature for iron ore without fusion and using solid carbon as reducing agent is about 875° to 1,025°C., the most desirable temperature being about 950°C. The heat conductivity of finely divided iron-coal mixture is low. Also the reduction reaction in such changes are endothermic. These conditions make rapid heating of the charge almost the first essential factor in the design of a high output furnace. The muffle furnace is less practical than the direct-heated type for the reduction of iron ore on a large scale.

The large-scale experiments are carried out in continuous rotary gas-fired kilns. The kilns are built with sections of different diameters. The charge of ground iron ore and coal enters the upper end of the kiln and passes rapidly in a thin layer through the preheating section. During the preheating period of 15 to 20 min., the volatile matter of coal and the moisture in the charge are driven off and the charge enters the enlarged section at approximately 900°C. with 3 to 5 per cent iron reduced. In this section the charge is in a layer of 13" to 15" deep. Maintained at a nearly constant temperature of 900° to 1,000°C., the charge remains in this section for about 1 hr. during which period the reduction of the iron oxide is complete. The reduced product is discharged continuously through a pipe into a cooling chamber below the furnace. The cooled product contains a large quantity of excess carbon and coal ash which is removed by means of a magnetic separator. The concentrate usually contains less than 1 per cent carbon and 65 to 90 per cent iron according to the grade of ore treated. The experiments indicate that rotary kilns give consistently good results<sup>5-13</sup>.

The *Bureau of Mines* have also carried out experiments, using natural gas as a reducing agent<sup>14,15</sup>, with ore containing about 50 per cent iron, 10 per cent silica and 3 per cent alumina. A large part of the investigation is devoted to the development of a suitable reducing gas from natural gas. The following conclusions have been reached : (i) In a 25,000 lb. charge, 80 per cent of the iron ore is reduced to metal in 13 hr. ; (ii) a higher degree of reduction is not obtained because the amounts of carbon dioxide and water vapour present retard the rate of reaction, and because the endothermic nature of the first reaction reduces the temperature below the optimum ; (iii) sintered ore is not reduced as rapidly as unsintered ore ; (iv) discharge of the sponge iron is difficult because of sticking ; and

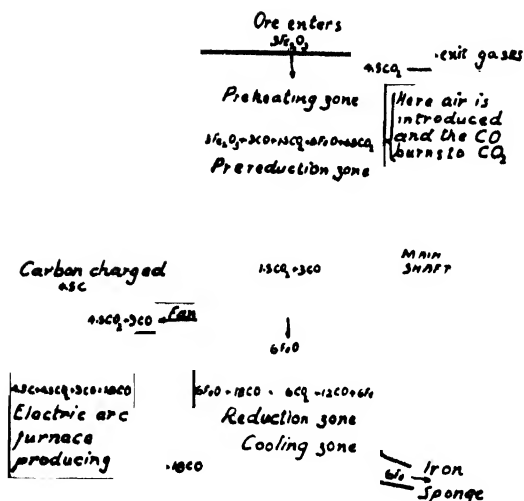


FIG. 1 — DIAGRAMMATIC REPRESENTATION OF WIBERG PROCESS.

(v) the sponge, containing 80 per cent iron in the reduced state, is easily melted in an electric furnace.

Producer gas was successfully used in a multiple-hearth furnace<sup>16</sup>. Republic Steel Corporation has built a multiple-hearth furnace using coke-oven gas<sup>17</sup>. Hydrogen gas was tried in what is called bubble-hearth process<sup>18</sup> but the process was found unworkable.

## 2. Swedish Processes: Wiberg Process —

The theoretical basis of a suitable process for the manufacture of sponge iron was laid down in 1918 by Prof. Wiberg. In principle (FIG. 1) the method is as follows: The ore, either in sintered form, or in the form of uncrushed ore of high iron content, is charged into the furnace, which works on the counter-current principle.

In the lower part of the shaft, carbon monoxide is passed in at a temperature of  $900^\circ$  to  $1,000^\circ\text{C}$ . The gases passing up the shaft reduce the ore to iron, and the carbon dioxide content of the gas rises to 25 to 30 per cent with respect to iron, an equilibrium between ferrous oxide and carbon dioxide being set up. Three-fourths of the gas is sucked out by fans into an electric arc gas producer, in which it is reduced to carbon monoxide. The reaction  $\text{C} + \text{CO}_2 \rightleftharpoons 2\text{CO}$  being endothermic, heat must be supplied, and this is done by electrical energy. The remaining gas (one quarter) passes further up the shaft where an air intake causes it to be burnt, thus heating up the ore to  $900^\circ$  to  $1,000^\circ\text{C}$ .

A furnace capable of producing 10,000 metric tons per year was erected in Soderfors by the *Stora Kopparberz Berzslag Company* in 1932. Fig. 2 shows a sketch of the furnace. The ore or sinter is laid in at (1). The shaft is lined with fire-bricks and has a wall thickness of about 900 mm. This is surrounded by a sheet-iron cover. The gas is laid in at (2) through a ring conduit, and then through a number of narrow twyers in the shaft (3). The gases are led out through 4 exits (4). The air for the combustion in the upper part of the shaft enters through 12 twyers and the used gases finally leave by the chimney (6). The chimneys have valves to regulate the pressure in the shaft. The lowest part of the shaft is surrounded by circulating water (7). The sponge leaves the outlet (8) into the container (9). The gases from (4) pass through the circulating fan (10) which, revolving at 2,950 r.p.m., could deal with 110 cu. m. per min. at 360 mm. pressure and  $800^\circ\text{C}$ . The temperature and pressure conditions in the furnace installation is illustrated in Fig. 3.

The gas producer (FIG. 4) is lined in the same way as the shaft, the outer cover being of 18-8 Cr-Ni steel. The producer has three pairs of Soderberg electrode, 300 mm. diam., passing down through a collar, each electrode being coupled to its own single-phase transformer. The transformers are of 400 kW. at 55 to 90 volts. The carbon is fed in through (12) from the carbon container (13) of 14 cu. m. capacity. The dust and ash have to be shovelled out at the bottom.

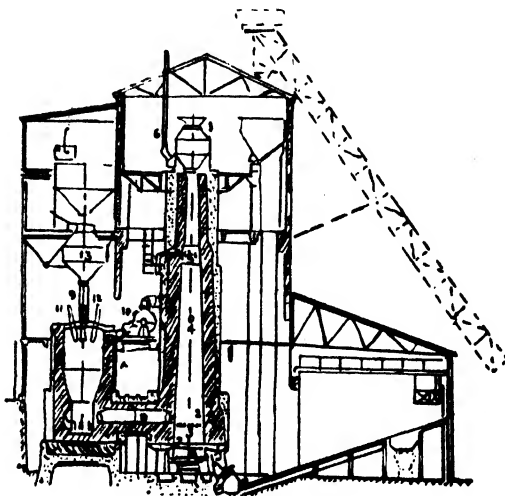


FIG. 2 — SPONGE IRON FURNACE AT SODERFORS.

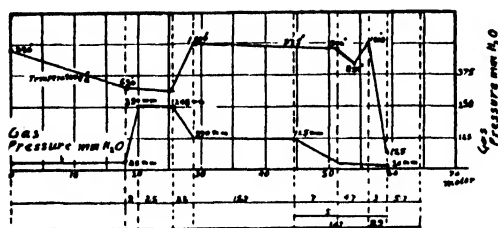


FIG. 3 — TEMPERATURE AND PRESSURE CONDITIONS IN A SPONGE IRON INSTALLATION.

With a given size of the furnace, the output is dependent on (a) temperature and composition of in-going gases; (b) speed of gas circulation; and (c) quality of the ore. The correct composition of the gas is easily achieved and its temperature can be closely defined within practical limits. Increase in speed of circulation increases output. Above a certain limit, however, the amount of carbon dioxide in the gases entering the producer is reduced, since the ore is not sufficiently rapidly reduced for the carbon dioxide to be increased to the required theoretical value of 30 per cent and the electricity and charcoal consumption per ton of reduced iron ore automatically increases. The suitability of the ore can be increased by pretreatment<sup>19-23</sup>.

According to Ramoeyer<sup>24</sup>, the Wiberg process is the only one in successful operation at present, but it has three disadvantages. It cannot use the fine concentrates just as they come from the magnetic separator; secondly, the operation has to be carried out at such a high temperature that sticking difficulties are encountered; and thirdly, regeneration of carbon dioxide to carbon monoxide is not very satisfactory. The use of hydrogen under pressure is advocated as a reducing agent. The reduction of magnetite to iron with hydrogen should not be carried out above 1,100°F., otherwise ferrous oxide will be formed which is difficult to reduce.

These considerations have led to the development of the Brassert process, in which coke-oven gas is used as the source of hydrogen. The coke-oven gas is desulphurized, preheated to 1,200° to 1,300°F. and passed into the bottom of a vertical Herreshof furnace into which ore is fed at the top. The finely divided ore passes down over a series of disc plates to the slotted grate at

the bottom through which the hydrogen passes upwards. By bubbling the gas through the fine ore, the latter is made to flow along the channels and from one plate to another as if it were a liquid. To prevent reoxidation, the hot reduced fines are passed directly to a briquetting press designed to make corrugated slabs of compressed sponge iron.

3. *German Processes: The Krupp & Krupp-Renn Process* — The direct reduction of ore is carried out in a rotary furnace; the iron is obtained in the form of compact lumps containing but little carbon. On leaving the furnace the lumps are embedded in a pasty slag, from which, after cooling, they are separated by a crushing process. All sorts of low-grade fuels, such as coke screenings, fine coal and semi-coke residues, can be used in the furnace; the recovery of iron in the lumps amounts to 90 to 96 per cent, depending on the ore and the mode of operation.

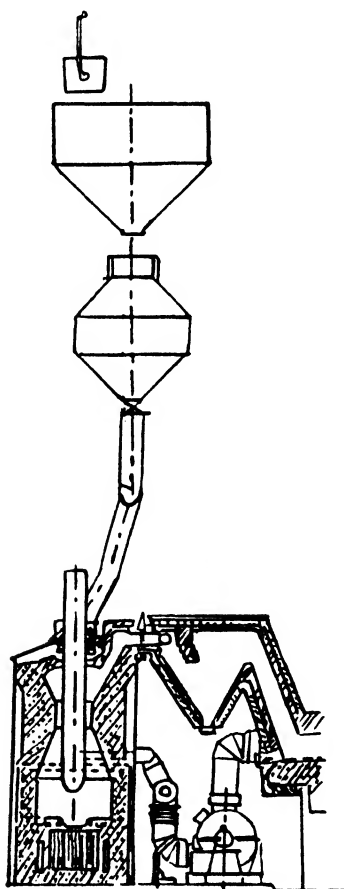


FIG. 4 — SINGLE-PHASE GAS PRODUCER.

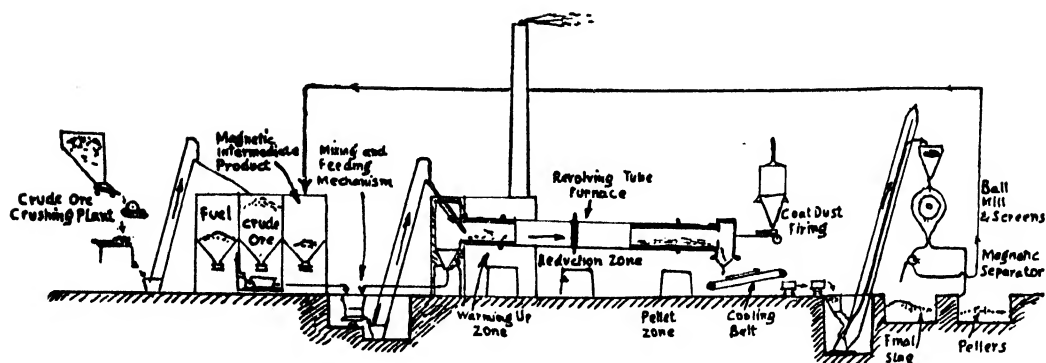


FIG. 5 — OPERATIONAL SEQUENCE IN THE IRON PROCESS.

Most of the phosphorus from the ore passes into iron, but manganese goes largely into the slag. The sulphur content of the lumps depends on the basicity of the slags. When treating rich ores, it would appear economical to aim at the production of low-sulphur lumps by making suitable additions to the charge, and then to use lumps directly for steel-making. With poor ores, and particularly those high in silica, it is better to make no additions but to use the process simply as a preparatory treatment and then charge the lumps into a blast furnace<sup>25-27</sup>.

The operational sequence of the Renn process is set out schematically in Fig. 5. As opposed to the well-known processes for the production of iron by means of the revolving-furnace process in which, as a rule, the iron is produced as a sponge or liquid pig iron, the essential feature of the Krupp-Renn process lies in the special function of the "pallet-zone".

Fig. 6 shows the bottom ends of a Walz furnace (used for the distillation of zinc and lead) and a Renn furnace in sectional elevation.

The Krupp-Renn process definitely makes smaller demands as regards the composition of the slag than does the normal smelting process. The process only requires a slag which is semi-soft at 1,200° to 1,300°C.

The physical properties of the fuel, such as screen size and resistance under pressure, play no part as the fuel is finely ground and mixed with the ore. Fuel with a high ash content causes no difficulties and is preferable to low ash material if it can be obtained at lower price.

The pellets obtained by the Krupp-Renn process are practically slag free and vary in size from 1.5 to 50 mm.

There are three principal applications of the Renn process (Fig. 7) :

- (i) The direct production of steel ;
- (ii) the enrichment of a poor quality ore, especially one with a high silica content ; and
- (iii) the production of non-ferrous metals, for example, nickel, cobalt, copper, silver, gold and platinum metals.

The direct production of steel without the use of the blast furnace is particularly suitable in cases where a supply of ore is available, but suitable coke for blast-furnace smelting is not too plentiful.

Briquetted iron ore is used in another German process<sup>28</sup> in which carbon monoxide, hydrogen or other suitable gas brings about the reduction in a shaft furnace. The ore reaches its highest temperature about half way down to the furnace, and is cooled down almost to the atmospheric temperature by

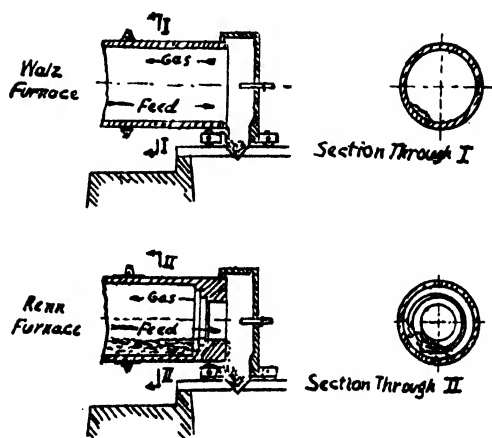


FIG. 6 — BOTTOM ENDS OF WALZ AND RENN FURNACES.

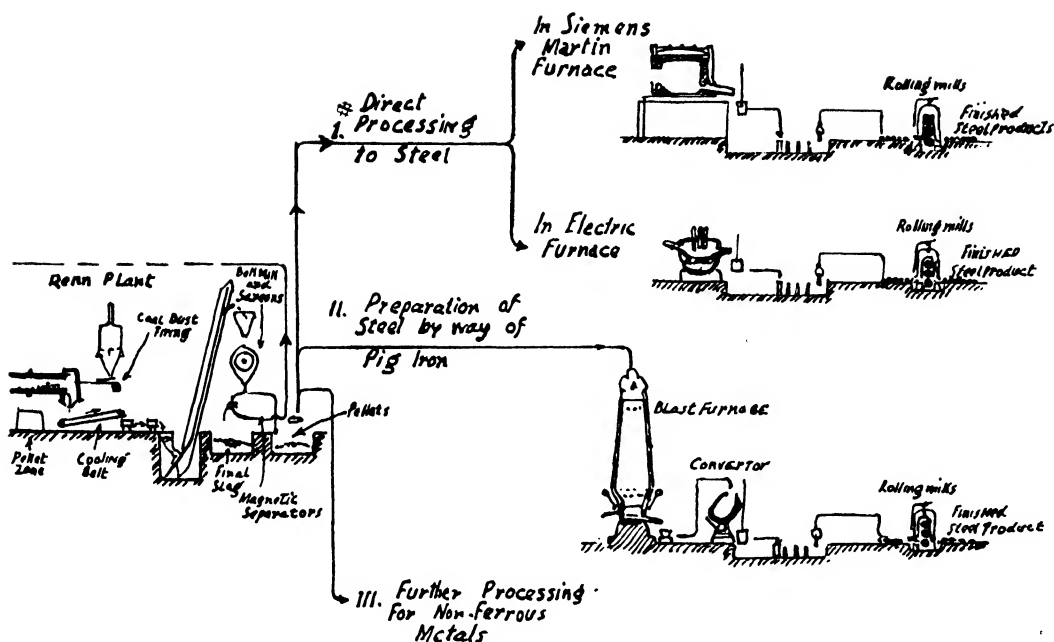


FIG. 7 — PRINCIPAL APPLICATIONS OF RENN PROCESS.

a cold blast of reducing gas admitted at the base of the furnace, wherefrom the sponge iron is extracted.

*Norsk-Stahl process*<sup>29</sup> uses a mixture of carbon monoxide and hydrogen as a reducing agent. The equipment consists of three vertical ovens for preheating, reducing and cooling the charge and apparatus for regenerating the spent gases.

Other important processes are: *Hornsey process* worked at Chesterfield<sup>30</sup> and *Lorain and Ohi*<sup>15</sup> in which rotary kilns or retorts are employed. The *Smith process* is carried out in vertical ovens or retorts, similar to coke ovens in design. The crushed ore is mixed with carbonaceous material and charged into the oven where it is heated and cooled by means of horizontal flues. It is preheated in the upper part of the oven by waste gases which leave the stack at about 400°F. The charge then enters the reduction zone where temperature ranges from about 1,600° to 2,000°F. The charge is subsequently cooled by the in-coming air for combustion in the heating flues, and discharged below 250°F.<sup>31</sup>

#### Economic Aspects<sup>32</sup>

The economic success of a direct process depends on: (i) whether it can produce a finished product which requires no remelting

before marketing at a fair price in competition with other metals; (ii) whether it can produce a material at as low a price as steel scrap; and (iii) whether the manufacturers can obtain for sponge iron a price better than that of steel scrap. The direct process has a few disadvantages compared with blast furnace. It cannot easily flux off sulphur (Bush and Schout have investigated the production of low-sulphur sponge iron<sup>11,33</sup>). Another disadvantage is that the material is readily oxidized and is difficult to handle during melting operation.

#### Summary

This report deals with the survey of the existing literature on the manufacture of sponge iron.

Methods of manufacture developed by the *United States Bureau of Mines* are enumerated, their experiments on continuous rotary gas-fired kilns are dealt with in detail.

The Swedish *Wiberg process* is described at great length. Given cheap electric power supply, this process is the most successful one.

The German *Krupp-Renn process* has been worked very successfully in Germany. In this process fuel with high ash content causes no difficulties and is preferable to low



ash material. Work carried out in other countries is also referred.

The economic aspects of the direct process are discussed.

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# Obituaries

## SIR ARDESHIR RUSTOMJI DALAL

(April 1884–October 1949)

WE REGRET TO RECORD THE DEATH, ON October 8, 1949, of Sir Ardeshir Rustomji Dalal at Bombay.

Sir Ardeshir was educated at the Elphinstone College, Bombay, and later at the Cambridge University. He qualified himself for the Indian Civil Service and served the Government of Bombay in various capacities and was for sometime Secretary, Department of Education, Health and Lands, Government of India. In 1943 he was appointed a Member of the Viceroy's Executive Council in charge of Planning and Development, in which capacity he served till the end of 1945. During the latter period he was President of the Governing Body of the *Council of Scientific & Industrial Research*.



SIR A. R. DALAL

More recently, as the Chairman of the Reviewing Committee set up to review the work of the Council, he was responsible for formulating far-reaching recommendations on the development and utilization of scientific research in India.

Sir Ardeshir was one of the principal signatories to the well-known 15-year Bombay Plan for the industrial development of India, a plan which is regarded as a landmark in the history of post-war economic planning.

After retiring from the Indian Civil Service, he joined the *Tatas* as Director and partner, and was one of the leading industrial magnates of the country. Dignified and broadminded, he represented in the larger national life of the country the great traditions associated with India's leading Parsis.

## MR. C. C. GHOSH

WE REGRET TO RECORD THE DEATH, ON August 20, of Mr. C. C. Ghosh, retired Deputy Director of Sericulture, Bengal, at the age of sixty-five.

Born of a middle class family, Mr. Ghosh developed a keen interest in entomology and from a technical assistant in the Pusa Agricultural Research Institute, he rose to the post of the Entomologist to the Government of Burma, in which capacity he made valuable contributions to the organization of sericulture industry in Burma. His contributions to the development of sericulture in Bengal, during nearly a decade prior to his retirement from service, are well known.

Among his many publications, mention may be made of *Insect Pests of Burma*, which is accepted as a standard work in economic entomology, the *Silk Industry of Japan* and *Silk Production and Weaving in India*. He was Chairman of the Silk Research Committee of the *Council of Scientific & Industrial Research*.

# REVIEWS

**Forest Pathology**, by John Shaw Boyce (McGraw-Hill Book Company Inc., New York), 1948, Second Edition. Price \$6.

THE BOOK IS BASED ON THE RESEARCHES on forest and plant pathology contributed by about 300 workers all over the world covering a period of three quarters of a century. The first edition was published in 1938 which underwent 4 impressions in a comparatively short period. The first book on this subject was published in 1931 under the title *An Outline, of Forest Pathology* by Dr. Earnest E. Hubert. Dealing with the same subject is a third book, *Pathology in Forest Practice*, by Professor Dow Vawter Baxter published in 1943.

Forest pathology, which principally deals with the diseases of woody plants of forests and also takes into account the decay of timber and deterioration of forest products, is a daughter science of plant pathology and applied mycology. But the extension of its scope to forest products has made it more attractive and important than the nucleus from which it developed. Like every applied science, the field of application of forest pathology is ever expanding. This is shown by the growing demands of a treatise like this and the publication in the last 12 to 15 years of 3 well-written books and a large number of subsidiary texts on decay of timber and forest products, monographs on tree rusts and other fungi connected with tree diseases.

The fact that the first edition of Professor Boyce's book underwent 4 impressions during the war time is significant of its popularity and of the demand by increasing numbers of workers in this field for information on more recent developments. A large amount of new information has accumulated during the past 10 years, mainly on the exploitation of timber resources and intensive utilization of forest products for purposes of war and this had to be incorporated.

The book is compiled in 21 chapters, 2 appendices (containing a description of fungicides and a list of common plant names with their scientific equivalents), and an index. The book can be broadly considered

under 7 parts, namely general mycological text, non-infectious diseases, seedling diseases, pathology and tree diseases, decay of wood and wood products, principles of disease control, and appendices and index.

The first 3 chapters consist of introduction, discussion on the causation of fungal diseases and a simple taxonomic description of different groups of fungi. Chapter IV on non-infectious diseases discusses pathological symptoms produced by high temperatures (sun scald), low temperature (frost injury, frost ring formation, frost canker, frost scars, etc.), water deficiency (drought, heat cracks, winter drying, etc.), water excess, nutritional defects, injury due to industrial process (smoke and smelter fumes injury), salt spray, mechanical injury (fire scars, snow storms, hailstones and lightning injury, etc.), and provides a complete bibliography to the recent works of Swingle (1944), Hepting (1945), Jackson (1945), Hansborough (1947), Hawboldt (1947) and Pamerleau (1944). The seedling diseases have been considered under 2 categories, namely non-infectious and fungus diseases. Under non-infectious diseases, the above factors have been considered from the point of view of protection. Under fungus diseases, damping off, root rot, snow blight, moulding, smoothening, spots, etc., due to the attack of fungi of lower orders (Phycomycetes and Fungi Imperfecti) and Ascomycetes and a few Basidiomycetes have been discussed. For controls of seedling and nursery diseases, selection of disease-free seeds, proper planning of nursery and selection of nursery sites have been given the utmost importance. Although the list of causal agencies is not complete and reference has been made to pathogens known in the United States of America, the items of control practically cover the whole range of such diseases. The root disease due to the shoe-string fungus (*Armillaria mellea* (Vahl.) Quel.) has received adequate recognition (chapter VI). Contributions on the pathology of root and buttrot fungus (*Fomes annosus* (Fr.) Cke.) from the United States of America and Europe are mostly of recent years (1930 to

1946); the readily accepted theory of root wounds and frost injury in the butt region has been discussed with a few remarks on control methods. A short description of root diseases due to other organisms of the family Basidiomycetes, Ascomycetes, Phycmycetes, Fungi Imperfecti and viruses (Phloem Necrosis of American elms—Swingle 1942, Tucker 1945) have been given. A short account of mycorrhiza has also been included.

The foliage diseases have been described in 2 parts: hardwoods (chapter VII) and conifers (chapter VIII). The former include leaf spots, tar spots, twig blights, powdery mildews, sooty moulds, rusts, blisters, flower and fruit rot, deformations, scab, and canker of tender shoots caused by various pathogenic fungi including rusts, Ascomycetes, Fungi Imperfecti, etc., a short but comprehensive account being given.

Under the latter are included distribution, pathology and biology of leaf blights and needle casts due to various ascomycetous fungi such as cedar-leaf blight due to *Keithia thujina* Durand, needle cast of Douglas fir due to *Rhabdocline pseudotsuga* Syd., needle cast of balsam fir due to *Hypodermella nervata* Darkar, needle cast of pines due to *Lophodermium piniastris* (Schr.) Chev. *L. juniperinum* (Fr.) de Not., *Hypodermella concolor* (Dearn.) Darkar, of jack and lodgepole pines have been given, besides several others, under miscellaneous foliage diseases. Under needle rusts distribution, life-cycle, pathogenicity of cedar and juniper needle rust (15 species of *Gymnosporangium*) have been considered in detail because damage to the pomaceous hosts is often serious in the west coast, north, central and Lake Region States of U.S.A., and in the provinces in the north, central and western Canada and valuable orchard trees are attacked by "cedar apple" rusts. Control measures include spraying the orchards and isolation of alternate hosts. Besides *Gymnosporangium*, about 70 species of needle rusts, most of them being heteroecious species, have been described hostwise such as Pine Needle Rusts (*Melampsora*, *Melampsorrella*, *Uredinopsis* and *Milesia*), Hemlock Needle Rusts (*Pucciniastrum*), and Larch Needle Rusts (*Melampsora*). Also included in the list are unrelated species referred to by form genera *Peridermium*.

The stem rusts of conifers which cause "witches'-brooms", swellings, blisters and galls of stem and cause die-back or kill young

trees outright are of greater economic importance and have received detailed treatment in two sections. Distribution, biology and pathology of 10 heteroecious stem rusts, except in case of Woodgate Gall Rust, the identity of which is not known, and of Western Gall Rust (*Cronartium harkensii* (Moore) Menecke) which is suspected to be identical with Eastern Gall Rust (*Cronartium cerebrum* Hedge. and Long = *Cronartium quercuum*), have been described in detail. Nothing could be more welcome to students of forest pathology than the complete and up-to-date supplementary information at the end of each chapter. One chapter has been allotted to the White Pine Blister Rust in the U.S.A. which is introduced from Europe. The amount of damage it caused to the pines, endemic as well as introduced, made the problem of paramount importance to the States for a quarter of a century. References have been made to the works of about one hundred workers in the States and in Canada, and a direct method of control has been successfully worked out. The White Pine Blister Rust, the Chestnut Blight Disease and Dutch Elm Disease have strengthened the hands of American phytopathologists in enforcing quarantine laws.

This is followed by 2 chapters on the stem diseases of conifers and hardwoods. In the former, canker due to Ascomycetes such as canker of pines due to *Tympanis species*, canker of larch due to *Dasyctypha willkommii* (Hart.) Rehm and other species of *Dasyctypha*, and due to Fungi Imperfecti such as *Macrophoma species*, *Phomopsis lokoya* Hahn have been described and symptoms and etiology detailed. These diseases attack trees growing under unfavourable conditions in the virgin forests of the United States; the descriptions of these parasites, therefore, have been given from the point of view of the American workers. None of these diseases, however, has been recorded in India.

In dealing with the stem diseases and cankers of hardwoods, which are of more serious consequence than those of conifers, the author has recorded many new diseases which are difficult to diagnose, and still more to control unless their etiology is known. Protective measures, which were merely suggestions, have now become definite with the increasing knowledge of the etiology and biology of the organisms. This chapter has considerable additional information than the previous edition.

Amongst the Fungi Imperfecti, *Dothichiza* and *Septoria* cankers of oak and chestnut, *Sphaeropsis* cankers of oak; and amongst the Ascomycetes Black Knot of cherry (*Dibotryon morbosum* (Schw.) T. & S.), *Didymosphaeria* canker of alder, *Cytospora* canker of willow, *Neofabraea* canker and *Hypoxyton* canker of poplar and *Eutypella* canker of maple have been described. *Nectria* canker (*Nectria galligena* Bres.) of yellow poplar and magnolias and Beech Bark Disease (*Cryptococcus fagi* Baer. followed by *Nectria cocinea* var. *faginata*) with symptoms and etiology have been described in detail. Lastly, the Chestnut Blight or *Endothia* canker of chestnut has been described. This section has been presented in a somewhat more condensed form than in the previous edition. This fungus was formerly considered a dangerous parasite of forest *cum* horticultural plants of American importance. The Chestnut Blight Disease of America is now a wider problem for the geneticists and tree pathologists as it affects vast regions of Europe and Asia and the information regarding all aspects of the problem will be useful for workers in this field.

Under stem diseases, non-infectious and infectious galls have been discussed in 2 chapters. Short accounts of dieback of hardwoods due to *Nectria cinnabarina* (Tode) Fr., of pines caused by *Cenangium abietis* (Pers.) Rehm., of butternut by *Melanconis jugulandis* (E. & E.) Graves and of other miscellaneous hardwoods due to various species of Fungi Imperfecti have been given. A complete description of Dutch Elm Disease has been given. For protection of elms, sanitation against the bark beetles that spread the fungus, spraying of technical DDT dissolved in fuel oil or xylene with emulsifying agents has been recommended.

A concise description of American mistletoes with a key to the American species of *Arceuthobium* together with the host list has been given in chapter XV. Protection methods have been discussed in detail from the pathological and silvicultural points of view for all types of forest including nursery stocks and virgin stands. At the end of the chapter some aspects of biological control have been discussed and a few enemies of mistletoes have been referred to.

Further elaboration of stem diseases due to various hymenomycetous fungi which are regarded as facultative parasites and for

brevity termed "decay" opens chapter XVI which introduces timber pathology. The descriptions are concise but a full treatment has been given under each section which follows.

The importance of the isolation of "decay" and the examination of cultural characters for the diagnosis of "decay" (Campbell 1938; Davidson, Campbell and Vaughn 1942; Robak 1942) have been emphasized. Special media which accelerate the formation of sporophores (Badcock 1943), oxidation tests with gallic and tannic acids in malt agar media and the guaiacum tests are useful recent additions.

Under the section "Control of Decay", methods of scaling and estimating "decay" in trees and logs have been discussed and the methods to calculate the volume of rot and sound timber in stands and timber have been given. This is a very important pre-requisite for those engaged in the timber trade. The author has considered decay in various American softwoods such as western conifers which include Douglas fir due to *Fomes pini*, *Fomes laricis*, *Polyporus schweinitzii* and *Fomes roseus*; sitka spruce due to *Fomes pini*; western hemlock due to *Echinodontium tinctorum*, *Fomes emosus*, *F. applanatum* and *F. pini*; white pine and lowland white fir due to *E. tinctorum*; red fir also due to *E. tinctorum*; western white pine due to *F. pini*; ponderosa pine due to *Polyporus anceps* and incense cedar due to *Polyporus amarus*. Data from various Canadian and American laboratories and references to well-known workers such as Bier, Foster, Salisbury, Weir, Lewis, Hubert, Englarth and the author himself have been included. Importance of such timber like sitka spruce which was used for aircraft during the second World War, red fir, ponderosa pine, etc., not mentioned in the earlier edition has been stressed and decay tests included. Formation of the top rot of hardwoods and its relation to the incidence of wounds and rotten branches and the data from the works of Davidson and Campbell (1943) on Black Cherry Rot on the estimation of cull defects are recent additions.

The largest number of contributions on stem and timber decay in recent years is mainly from the forest products laboratories of Germany, Sweden, England, Canada and U.S.A. and also from the laboratories under many industrial concerns processing timber and wood products and are referred to in chapter XVII. The author has given a

complete but concise description of some of the important American timber-destroying fungi. Besides short descriptions of about 80 species of hardwood and softwood decay, estimates of losses due to decay, both in use and storage, have been included. The section on dry rot has, however, been disposed of with short remarks on *Poria incrassata* (B. & C.) Curt. with a mention of *Merulius lacrymans* (Wulf.) Fr. The bibliography on the rots is complete.

The last 3 chapters are important from the point of view of timber industries, lumbering, utilization of forest products, etc., and subjects dealt with in sections under different categories are: Deterioration of Dead Timber (chapter XVIII); Deterioration of Forest Products—Decay (chapter XIX); and Deterioration of Forest Products—Sap-stain (Chapter XX). The author discusses the methods of salvage of killed trees, decayed and stained wood and wood products. The sound principles of lumber industry are based on secondary operations and salvage of the so-called waste material. Insect-killed, fire-killed, wind-thrown timber and slash, etc., add to the losses.

Biotic factors are responsible for tremendous losses of timber resources in tropical forests and the information given in these 3 chapters should prove very useful to our foresters, lumbermen and industrialists. Fire, insect and wood-rotting organisms are considered as natural enemies of the tropical forests. We have no adequate means to stop them, but proper logging of insect-killed and fungus-decayed material would certainly save a fortune provided we are able to work up the proper schedule of deterioration that sets in at different stages so as to ascertain how long such timber could be left in the coupé at the time of the year when transport to the mills is not practicable. Fire is the greatest curse of tropical and subtropical forests. We have no means to calculate the actual amount of loss to forests caused by this agency, but the estimated loss runs into many crores of rupees.

From the utilization point of view, the natural resistance to decay due to the by-products of plant metabolism such as tannin, volatile chemicals, colouring matters, toxic oils, resinous exudations (non-toxic but prevent penetration of wood-destroying fungi), etc., have been considered. Under the section of decay of pulp wood and pulp, methods of hygienic storage, proper piling and chemical treatment to protect material

for pulp have been briefly discussed. The loss to our pulp material (bamboo in particular) is very high due to the fact that sap-stain is active in the monsoon season for 4 to 6 months. Storage is a difficult problem for the paper industries. To save the tremendous wastage, chemical protection of pulp material is essential.

Much work has been done by American workers on sap-stain since the publication of Professor Boyce's book on forest pathology in 1938. Stains on wood products, which were regarded as minor defects in timber, especially the blue stains of softwoods, are now considered to be one of the major handicaps to the marketing of finished wood products. Information available on the control of sap-stain (Schaffer and Lindgreen 1940, Hartley 1945, Schaffer 1946) has been given.

Under the section "Decay of Structures", fungi causing decay of building and other permanent structures, and methods to prevent decay have been summarized. The section on wood preservation has been unusually abridged but a complete bibliography to the subject has been annexed.

The author discusses the fundamentals of forest-disease control in chapter XXI under 2 categories, namely the control of native diseases and the control of introduced ones. The basic principles involved being different in the two cases, the methods evolved have been different. With the native diseases, sound silviculture, selection of sites, adjustment and judicial composition of the crop at the time of restocking, preference to mixed in place of pure stands in the second rotation, or second cut stands, selection of seed, acclimatization of the species when introduced from one region to another, and lastly, the breeding of forest trees to improve resistance have been considered and the merits and demerits discussed. The attempts to introduce exotics in the place of susceptible native species have been condemned. Introduced diseases are infinitely more destructive and require more expensive direct measures. The author has quoted glaring instances of half a dozen pests introduced into the United States during the last 50 years. The lack of judgement in the initial stage to estimate the harming capacity of potential parasites and delay in taking action against them have given them time to establish themselves on native American hosts. The slow method of investigating and cruising (estimating) the diseases

at the time when they are first noticed have also been criticized. Finally, the principles governing control of introduced parasites have been summarized and the views of Ditwiler (1929) and Fracker (1937) have been put forward. Public opinion has to be roused in support of effective sanitation and destruction of diseased stock, and the need for passing legal enactments when persuasive methods fail, has been emphasized.

Although the methods described relate to the checking of pests, both endemic and introduced, of native American trees, the basic principles discussed are applicable to any country faced with similar situations. We have fortunately no record of an introduced tree disease that has developed into dangerous stage. The diseases of our evergreen and deciduous hardwoods and of the conifers in the Himalayas so far recorded are endemic, and endemic diseases never develop to dangerous proportions. Furthermore, against native parasites direct control is rarely necessary or justifiable. We have a large number of resistant hardwood species. As regards conifers, the Himalayan species, though few, are sufficient for our home requirements, and except as a trial on an experimental basis, there is little need to introduce exotics in India.

During the last world war, it was difficult to safeguard any country against the introduction of new diseases. We have records of *Trametes serials* and *Poria monticola* reaching our shores in a consignment of sitka spruce and Douglas fir timber. There is every likelihood that other parasites have invaded the country through agricultural and forest products. Professor Boyce's remarks on quarantine regulations as an effective barrier against new pests are particularly apparent.

This book, which is the only one of its kind, will be invaluable to students and teachers of forestry, the forester, the silviculturist and the private owner of forest land. It will be equally handy to the lumberman and to everyone interested in the timber trade. The complete bibliography is invaluable to students, as no other complete

and up-to-date reference list is available in this country.

K.B.

**Elements of Electronics**, G. Windred (Chapman & Hall Ltd., London), 1949, pp. 197. Price 15s. net.

MR. WINDRED'S BOOK ENTITLED "ELEMENTS of Electronics" covers, in an elementary fashion, a wide field of the science of electronics. The text is divided into 15 chapters and deals with the principles of electronic charge measurement, thermionic emission, conduction *in vacuo* and gases, photo-electric effects, thermionic and gas-filled valves and circuits, X-rays, cathode ray tube and circuits, television, sound film, electron optics, cyclotron and radio location. The book is entirely non-mathematical and as such should appeal to those interested in electronics but without sufficient mathematical knowledge. The author's method of exposition is lucid and this he has ably maintained right up to the end.

However, in attempting to provide simple explanations for all subjects, the author has been rather vague in several places. For example, in the explanation of the working of the split-anode type magnetron, the statement on page 35, "in the unelectrified or neutral state a piece of matter contains a definite number of negative electrons, such that two pieces of matter in this condition will experience no mutual electric forces" is confusing. So also is a statement made in the paragraph which follows. In Figs. 57 and 58, the output terminals shown have no bearing with the text and as such are misleading. The grid bias arrangement and its measurement by voltmeter "V" in Fig. 65 is also wrongly shown. There are a few printing mistakes in the book.

But these criticisms are of a minor character and do not in any way detract from the value of the book. The author deserves congratulations for writing so clearly on a subject of wide interest like electronics.

G. SANYAL

# Fibres from Peanut Proteins

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THE successful use of soyabean proteins and milk casein as raw materials for the production of artificial textile fibres led to the investigation of the suitability of groundnut proteins as another possible raw material for the same purpose. Experimental work on the production of obtaining fibre from groundnut proteins was carried out in the laboratories of the *Council of Scientific & Industrial Research* with promising results.

The *Imperial Chemical Industries* have succeeded in producing textile fibre from groundnut proteins on a commercial scale. It is of interest to review the developments in this field in view of the work carried out by the author.

Groundnut, or peanut as it is known in America, is grown in India, China, Manchuria, Nigeria, French Sudan, Dutch East Indies, Spain and Argentina. India produces 180,000 tons of peanuts a year. China and Manchuria together produce about as much, and the United States rank third among the producing countries.

The process of fibre production from casein, soyabean and peanut proteins is similar to that employed in rayon manufacture, and can be considered under four main heads; viz.: (1) preparation of proteins; (2) preparation of the spinning solution; (3) spinning of fibre; and (4) hardening and after-treatment.

## Preparation of Proteins

The first step consists in the preparation of the oil-free cake from the residue obtained after pressure-expulsion of oil, through solvent extraction. The final cake has the following average composition:

	%
Nitrogen	10.5
Oil	1.5
Water	8.0
Ash	4.5

Peanut proteins are made up of the two globulins, arachin and conarachin, soluble in saline or alkali solutions. Extraction of proteins from the oil-free cake is one of the most critical operations in fibre production and demands extreme care for ensuring uniform batches. One method of extraction consists in soaking the powdered cake (of definite fineness) in 5 to 10 per cent saline at 15° to 20°C. for 6 to 12 hr. The quality of the finished proteins is influenced by the clarity of the extract, and a Sharple's supercentrifuge is employed for securing the desired clarity. The proteins are precipitated by adding to the extract freshly prepared, dilute sulphurous acid solution, or by slowly bubbling sulphur dioxide through the extract at 15° to 20°C., till a pH of about 5 is attained. The precipitated proteins are filtered, washed and dried at 30° to 40°C. The relative ratio of salt solution to the cake is also important. Ten parts of salt solution are usually employed for extracting the protein from one part of the cake. High temperatures during extraction lower the yield. Rate of stirring, concentration of sulphur

dioxide, pH at precipitation, extent of washing and temperature of wash liquor, and sunlight are other factors which influence the physical properties of the proteins precipitated.

In another method, a dilute solution of sodium hydroxide (0.1 to 0.5 per cent) is used for extracting the proteins. The dissolved proteins are precipitated by sulphur dioxide at a pH of about 5. The use of lime has also been suggested<sup>3</sup>. The final product is cream-white in colour, and the yield varies from 20 to 30 per cent on the weight of the cake.

## Preparation of Spinning Solution

The second step in fibre manufacture is the preparation of a viscous solution of high solid content. On account of the tendency of proteins to form a gel at high concentrations, it is difficult to prepare solutions containing more than 20 to 30 per cent solids. The solutions are then aged and "ripened", during which reorientation and partial denaturation of protein molecules occur. Denaturation is controlled and is allowed to proceed only to the extent of eliminating side chains leaving the main long-chain molecule intact. Thomsan and Traill<sup>4</sup> have shown that at higher solution temperatures, lower concentrations of alkali are required to uncoil the globular molecules to a linear configuration and later break them down to short chains. A viscosity between 40 to 300 poises<sup>5</sup> provides a solution which can be spun into fibres.

To impart the necessary pliability, softness and elasticity to the finished fibre, it is desirable to incorporate certain plasticizers in the spinning solution. One per cent oleic acid, linseed oil acids, Turkey-red oil, sodium glycerol phosphate, sodium tartarate, glycol phthalate, ethyl glycollate, butyl tartarate, amyl acetate, sodium maleate and urea (in the order of their effectiveness) are a few of the plasticizers in use.

As strengthening agents (for increasing the tensile strength of the fibre), salts of aluminium, calcium, barium and, to a less degree, magnesium are recommended. While using calcium and barium salts it is necessary to use the sulphonates of alcohols, fats or aromatic radicals, with or without hexa-metaphosphate, to prevent premature precipitation of proteinates. Sodium lauryl sulphate and sodium oleyl sulphate are particularly effective. Of less value as strengthening agents are sodium silicate, sodium chromate, sodium tungstate and sodium stannate.

Technically, the preparation of spinning solution consists in soaking the proteins in water for 30 to 60 min., and grinding to a uniform paste. A small amount of alkali (1.0 to 1.5 per cent) is added slowly with continuous stirring when all the proteins go into solution. Plasticizers and strengthening agents are added and intimately mixed. The solution is then filtered in a stream-line filter and aged at 10° to 15°C. for 15 to 25 hr. The quality of different batches of the spinning solution depends upon the size of protein particles, rate of stirring, rate and order of addition of the additives and other factors.



*Spinning* consists in forcing the protein solution through spinnerets into an acid bath, and collecting the filaments on a bobbin as in the spinning of rayon.

The process may be conveniently described under two main heads:

**Precipitation** — Effective precipitants<sup>7</sup> are sulphuric acid, phosphoric acid and acetic acid (in 0.5 to 15 per cent concentration). Addition of small amounts of sulphates of calcium, aluminium and sodium and glucose to the bath provides a better control of the speed of coagulation and facilitates dehydration of the filaments. A small amount of formaldehyde is added to the bath for imparting strength to the fibre at an early stage.

**Spinning Technique** — The spinning solution, properly aged and freed from insolubles and air bubbles, is forced through fine nozzles dia. 10 microns) into an acid bath where the proteins are precipitated in the form of fibres, which are wound on a bobbin. Although horizontal baths are in general use, vertical baths may be more suitable. Stretching of the fibre during its precipitation is effected by pulling it through the acid bath over two glass pulleys revolving at different speeds. Stretching appreciably influences the physical properties of the finished fibre, unstretched fibres being brittle and weak.

When the spinning solution comes into contact with the bath solution, coagulation of the proteins occurs at the surface, leaving a core of uncoagulated solution within the fibre cylinder.

When the fibre is subjected to stretching, the rate of pull will be more on the coagulated solid layer than on the enclosed uncoagulated solution, i.e. a shear will be set up which brings about combing, alignment or orientation of the protein molecules. As the fibre leaves the acid bath (the length of the bath is about one metre), the enclosed solution has to be completely coagulated. It is obvious that to produce a good textile fibre, there should be a proper correlation between the speed of stretching and the rate of coagulation. The accompanying diagram (Fig. 1) illustrates the different speeds at the axis and circumference of the coagulating fibre. Spinning speeds up to 400 to 500 ft. per min. are generally employed<sup>8</sup>. Viscosity and temperature of spinning solution, speed of extrusion and stretching of the coagulated fibre, temperature, and composition of the acid bath, etc., are all factors which influence the final characteristics of the fibre.

The hardening of the fibre is brought about by a relatively long immersion (12 to 24 hr) of the tow (thousands of tiny strands coming from the spinnerets) in a formaldehyde bath maintained at a definite temperature, concentration and pH. The use of more than one bath with increasing formaldehyde concentration is recommended<sup>9</sup>. Other hardening agents<sup>7</sup> include acetaldehyde, croton aldehyde and butyraldehyde, but they are seldom used.

The amount of formaldehyde taken up by the fibre depends upon the pH of the solution. The presence of certain salts added to the bath also influences formaldehyde absorption.

Peanut fibre, unlike soyabean and casein fibres, forms a rod-like horny mass after the formaldehyde treatment and drying. To overcome this drawback, the hardened fibre is soaked in a hot solution of sulphurous acid in an organic solvent at 80°C. for 10 to 30 min., or in a solution containing sodium chloride and formaldehyde at

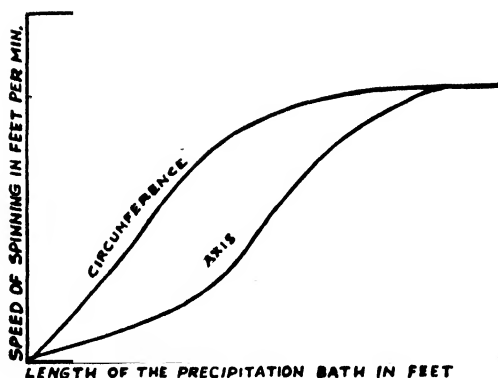


FIG. 1

35° to 40°C. followed by treatment with a 5 per cent batching-oil emulsion, or sulphonated oil and soap<sup>10</sup>. This treatment imparts the feel, pliability and other properties required for a textile fibre. The product, finally washed and dried under controlled temperature and humidity conditions<sup>11</sup>, resembles scoured wool and can be spun and woven by the conventional methods.

#### Properties of the Fibre

"Ardil", the commercial name given to the fibre produced by *Imperial Chemical Industries Ltd.*, is white to light-cream in colour and has a soft wool-like feel. The crimp in the fibre and the consequent inclusion of air in fibre bundles gives the fibre a warm woolly handle. All "Ardil" and "Ardil"-wool fabrics give the same thermal insulation as an all-wool fabric of the same thickness. It has a low tensile strength (15,000 p.s.i., and an extension at break of 80 per cent) and poor resistance to abrasion. It absorbs moisture like natural wool and its affinity for dyes is comparable to that of other synthetic fibres. It does not felt like natural wool and fur. Knitting yarns are prepared from "Ardil" mixed with cotton and rayon.

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# NOTES & NEWS

## Synthesis of Vitamin A

VITAMIN A IN A HIGH STATE OF purity has been synthesized at the *Warner Institute of Therapeutic Research*. Complete identity between the natural and synthetic product has been established. A method suitable for commercial production has also been worked out.

Starting with a  $C_{18}$  ketone, first described by Heilborn, a mixture of vitamin A acid and ester was obtained by the addition of 2 more carbon atoms. This was reduced to the vitamin, employing lithium aluminium hydride. The final product had a purity of 50-75 per cent, a concentration higher than any vitamin A concentrate commercially available up to now.

The synthesis of vitamin A in the form of its acetate, starting from  $\beta$ -ionone, has been achieved on a sufficiently large scale (*Chem. & Eng. News*, 1949, 27, 2106).

## Alcohol from Bassia Flowers

OPTIMUM CONDITIONS FOR THE fermentation of *bassia* flowers to yield industrial alcohol, together with a plant design, are described (*Ind. & Eng. Chem.*, 1949, 41, 1451).

*Bassia latifolia* flowers from C.P. and Berar containing, without hydrolysis 70 per cent reducing sugar as glucose, 5.9 per cent proteins (dry basis), and 18.4 per cent moisture, were used in these experiments. *Seagram yeast strain No. 90* (*S. cerevisiae*) employed in all the experiments was acclimatized by serial transfer through *bassia* medium. 20, 25 and 30 gm. of flowers per 100 c.c. of water were macerated and the material heated at 180°F. for 30 min. Conversion of sugar to alcohol was highest at 20 per cent concentration of the mash. A fermentation efficiency of 98.3 per cent after 72 hr. incubation and a plant efficiency of 92.6 per cent were obtained. The alcohol (95 per cent) yield was 5.32 gal. per 100 lb. of flowers, dry basis, in contrast to yields of 4.89 and 4.58 gal. for 25 and 30 per cent concentrations respectively.

To determine the effect of temperature and time on extraction

of sugar, whole flowers were extracted with agitation at room temperature and at 180°F. Cold extraction for 30 min. was found inferior to a similar hot extraction, as the former yielded 4.28 gal. as compared to 4.93 gal. 95 per cent alcohol in the latter case.

To augment the yield, various nutrients were added to 20 per cent mash which had been extracted for 30 min. at 180°F. Use of ammonium sulphate and ammonium phosphate at 0.2-0.6 per cent level yielded 5.05 gal. of alcohol 5 per cent. Urea was not found suitable as a nutrient.

A *bassia* flower distillery with a capacity of 2,000 gal. of industrial alcohol per day has been designed on the basis of this experimental work. The process consists of continuous automatic weighing of *bassia* flowers, water, and 5 per cent *bassia* stillage into a pre-cooker. *Bassia* slurry prepared in the cooker after a holding time of 20 min. at 180°F. is passed on for wet milling followed by a 30 min. batch cooking operation at 180°F. 5 per cent (by volume) of the mash is diverted for yeast propagation while the remainder is pumped continuously through a single-tube heat exchanger where it is cooled to the fermentation temperature of 80°F. Four 25,000 gal. fermenters provide adequate space for the 60-70 hr. fermentation cycle required by a 20 per cent *bassia* flower mash.

The distillation unit consists of a direct steam-heated beer stripping still, containing 18 perforated plates and 2 bubble cap plates, a beer heater which acts as a partial condenser, and an alcohol-vapour fed rectifying column, containing 30 bubble cap plates.

With *bassia* flowers at \$1.21 per cwt. f.o.b. the supplier, the cost of 95 per cent industrial alcohol manufactured by this process works out to \$0.372 per gal.

## Electrolytic Production of Azo-dyes

AN ELECTROLYTIC METHOD FOR the preparation of azo-dyes using an inexpensive iron anode in place of the usual expensive platinum anode has given satisfactory re-

sults in practice. The cell characteristics and the preparation of a few azo-dyes have been described (*Curr. Sci.*, 1949, 18, 291). The iron anode is not only cheap; it is corroded only to a negligible extent and does not promote nitrite oxidation. The yields of the dyes are also considerably improved.

The cell used is of the divided type, a porous alundum cup forming the cathode chamber and diaphragm. The anode is of heavy sheet iron, with the edges and corners rounded by filing. This anode is cleaned, heated to redness and chilled in water before being used; the reaction is found to be unsatisfactory without this treatment. The anode is an iron oxide anode; the thin layer of oxide formed helps in the passivation of the anode. A glass propeller-type stirrer is used for stirring.

On passing the current 3 times the theoretical length of time, a better yield of dye is obtained: 3.07 gm. or 61.4 per cent of the theoretical. The current efficiency, however, remains the same.

*Ponceau G*—This dye is obtained by the electrolysis of molar proportions of aniline, 2-naphthol-3, 6-disulphonic acid (disodium salt) and sodium nitrite in an alkaline electrolyte. The dye is formed in very poor amounts in neutral medium. 4.35 gm. R-salt, 1.16 gm. aniline, 0.90 gm. sodium nitrite and 0.50 gm. sodium hydroxide, in 150 c.c. of water are electrolysed at a current density of 5 amps./dm.<sup>2</sup> for 20 min. The dye is recovered from the anolyte by salting out. The precipitated dye is filtered at the pump, dried and weighed. Yield of dye: 1.15 gm. or 20.3 per cent of the theoretical. The colour on silk and wool is bright and of a good shade.

*Ponceau 2R*—4.35 gm. of R-salt, 1.50 gm. m-xylidine and 0.90 gm. of sodium nitrite are electrolysed in 150 c.c. of water, with the modifications mentioned above, at a current density of 5 amps./dm.<sup>2</sup>. The anolyte is coloured dark red at the end of the run. The dye is precipitated from solution on the addition of 25 gm. of sodium chloride with good stirring. The precipitated dye is filtered, sucked nearly dry and dried in an air oven at 80°C.

The yield of the dye is 95.5 per cent of the theoretical if the electrolysis is carried on for 2 hr. The quality of the dye is found to be satisfactory.

## Glass Sand Beneficiation

### Process

A TECHNIQUE OF ATTRITION GRINDING that makes possible the production of a high-quality glass sand from a low-grade sandstone has been described (*J. Amer. Cer. Soc.*, 1949, 32, 237).

The sandstone used is from Pottsville near Falls Creek, Pennsylvania. A typical sample analyses to:  $\text{SiO}_2$ , 98.75;  $\text{Fe}_2\text{O}_3$ , 0.12;  $\text{Al}_2\text{O}_3$ , 0.67;  $\text{TiO}_2$ , 0.10;  $\text{CaO}$ , 0.07;  $\text{MgO}$ , nil;  $\text{Na}_2\text{O}$ , nil;  $\text{K}_2\text{O}$ , 0.09; and ignition loss, 0.037, expressed as percentages. Petrographic examination of thin sections of specimens showed that about 60 per cent of the quartz was free and loose-grained; the remainder consisted of aggregates of quartzite, chert and iron-bearing minerals. The sand also contained about 1.5 per cent of a ferruginous clay as a coating on the grains.

To obtain a suitable sand for the manufacture of flint glass, the  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  content had to be reduced to about 0.025 per cent or less. The remaining impurities only needed to be made constant and it was not necessary to eliminate them.

The first beneficiation tests were started with impact grinding and washing. A sample of crushed rock, 40-50 mesh, was given washing and desliming treatments after which the sand contained 0.056  $\text{Fe}_2\text{O}_3$ , 0.061  $\text{TiO}_2$  and 0.07 per cent  $\text{Al}_2\text{O}_3$ . The remaining iron was present as a limonite stain on the particles and as discrete mineral particles with or without titania. This was followed by flotation and magnetic separation which were not found effective. A hot acid-leach was then tried with hydrochloric acid and stannous chloride. It was found that the iron content with 50 mesh sand could be purified to 0.023-0.026 per cent iron. The remaining iron was assumed to be held inside the quartz grains as it could not be removed. Acid-leach test with  $\text{SO}_4$  as the solvent also gave a similar product, although it took a longer time.

Large-scale beneficiation carried out with these methods proved complicated, economically unfeasible and unsatisfactory. Further study and microscopic work revealed that the sand had to be reduced in size by attrition grinding rather than by uncontrolled impact grinding. The pebbles had to slide over the surface of the sand grains to remove the limonite stain. The ratio of sand charge to pebble charge for maxi-

mum efficiency was found to be 1:1 to 8 for attrition grinding compared with 3 to 8 for impact grinding. The lower ratio permitted the reduction of sand by attrition to a certain size in one-half the time required to reduce the same amount of sand to the same fineness by standard impact grinding methods.

Attrition grinding resulting in a -150-mesh product (95 per cent) was found to be most economical and dependable. The beneficiated product contained 0.04 per cent  $\text{TiO}_2$ . Though this amount was not so low as the standard set originally, it was found harmless for the glass colour.

In the beneficiation plant described, sandstone from the quarry is subjected to primary crushing in jaw crusher and gyratory crusher and further reduced in a rod mill to -10 mesh. This material passes through a wet magnetic separator to the tube mills in a closed circuit with a bowl classifier, for attrition grinding to -150 mesh. This is freed from slime by a hydro-separator and passed on to a thickener, a filter and a drier successively. The water content of the product is less than 1 per cent.

### Contact Catalysts

CONTACT CATALYSTS MAY BE PREPARED by incorporating a suitable catalyst with a refractory carrier which is bonded by ethyl silicate. The preparation of contact catalysts by supporting a suitable catalytic agent on a refractory material is a common practice and usually the catalyst is prepared in pellet form. For example, a vanadium pentoxide catalyst supported on pumice pellets, bonded with sodium silicate, may be used to catalyse the oxidation of sulphur dioxide to sulphur trioxide. As a binder, sodium silicate has the drawback of impairing the refractory properties of the supporting material and also makes it difficult to obtain intricately shaped catalytic masses. If an organic silicate is used as the binding material, these drawbacks may be overcome. Ethyl silicate is generally chosen for this purpose. Suitable solutions can be prepared from ethyl silicate by hydrolysis with aqueous ethyl alcohol using hydrochloric acid as a catalyst. Since non-volatile inorganic bases are absent, the refractory properties of the supporting materials are unimpaired. Catalysts containing vanadium

pentoxide have been prepared in this manner.

Ethyl silicate containing up to 5 per cent of piperidine by volume may be slip-cast to give intricately shaped objects and after air-drying and firing between 700°C. and 1000°C., a hard, bonded refractory object is obtained, uncontaminated by inorganic bases.

By incorporating a suitable catalytic agent in the slurry during the above bonding process, the preparation of contact catalysts having complex shapes can be carried out, provided that the bonding process does not destroy the activity of the catalyst, or that the presence of the catalyst does not prevent the setting of the slurry. Instead of the direct incorporation of the catalytic agent in the mixture, a precursor which forms the catalyst on ignition may be added. Sillimanite blocks containing vanadium pentoxide and platinum catalysts have been prepared by this method. To prepare the vanadium catalyst, powdered hydrated vanadic oxide was mixed with the graded sillimanite. The block containing the platinum catalyst was prepared by suspending freshly prepared ammonium platichloride in the aqueous ethyl alcohol required to gel the ethyl silicate-piperidine mixture. The blocks were air dried and fired as described above.

After preparation, the catalyst blocks were granulated and loaded into a hard-glass combustion tube. Catalytic activities were assessed qualitatively by heating to redness and passing over the hot material a mixture of equal volumes of sulphur dioxide and oxygen. In both cases much sulphur trioxide was formed. A control experiment, performed with catalyst-free material, resulted in the formation of very little sulphur trioxide. The vanadium catalyst block was also found effective in the oxidation of ethyl alcohol. In the temperature range 100°-150°C., acetaldehyde was obtained whilst at 200°C., appreciable quantities of acetic acid were also formed. Tentative experiments indicated that the vanadium pentoxide catalyst is also effective in the oxidation of naphthalene to phthalic anhydride.

The above results indicate that vanadium pentoxide and platinum catalysts may be incorporated in sillimanite blocks bonded by an ethyl silicate and piperidine mixture, and retain their oxidative catalytic activity (*Chem. & Ind.*, July 1949, p. 500).

### Oxidizing Agent for Organic Compounds

GLYCOLS ARE OXIDATIVELY CLEAVED to carbonyl compounds by sodium bismuthate in an acid medium, the reagent thus behaving in a similar way to periodic acid and lead tetra-acetate; hydroxy acids are degraded to the lower aldehydes.

Two principal procedures are used: (a) The reagent is added to a solution of the glycol in glacial acetic acid and stirred or shaken at, or slightly above, room temperature, until the colour is discharged and a clear solution results (taking a few minutes or several hours); this procedure is also suggested as a convenient test for these classes of organic compounds. (b) The reaction is similarly carried out with the glycol in aqueous (purified) dioxan solution in the presence of phosphoric acid, the initially yellow-orange suspension being ultimately replaced by pure white bismuth phosphate.

The following substances have been oxidized and the products isolated (in parenthesis) in good yield (70-100 per cent): ethylene glycol (formaldehyde); diethyl tartrate (ethyl glyoxylate); *cis*- and *trans*-cyclohexane 1:2-diol (adipic aldehyde); hydrobenzoin (benzaldehyde); lactic acid (acetaldehyde); mandelic acid (benzaldehyde). The reaction has been successfully applied for the preparation of ethyl glyoxylate and adipic aldehyde, and also for pelargonic aldehyde and azelaic aldehyde acid from 9:10-dehydroxystearic acid (*Nature*, 1949, 164, 185).

### Corrosion Test for Plastics

A LABORATORY METHOD TO EVALUATE chemical resistance of various grades of plastics has been evolved by *Haveg Corporation*, U.S.A. (*Chem. Eng.*, 1949, 56, 98).

The weight loss test used for measuring corrosion in metals is not applicable to plastics which may lose weight due to evaporation of the plasticizer or gain by absorbing solutions, on account of their porous structure.

The new test procedure is based on 4 tests involving changes in the appearance of the sample and the solution (acid or alkali solution in which the specimen is kept immersed) and changes in weight and volume of the sample. All these data have to be considered together to arrive at a conclusion.

The tests are described with particular reference to *Haveg* (a proprietary product by *Haveg Corporation*) but are adaptable to all plastic materials. Further, in order to provide a semi-quantitative system of reporting results, a system of ratings on a percentage basis has been worked out. The final rating depends on the average of the 4 individual ratings for the 4 tests. This procedure also makes possible the comparison of results of different workers and prediction of field results with considerable accuracy.

The sample under test, cut into strips of  $\frac{1}{8}'' \times \frac{1}{8}'' \times \frac{1}{8}''$  strips, of known weight and original volume are placed in a  $\frac{1}{2}''$  dia. test-tube, 6" long. Stainless steel containers are advised for strongly alkaline solutions. For hydrofluoric acid and its compounds at elevated temperatures, *Haveg* 48 or a plastic tube made from pure phenolic resin is recommended. The end of the tube is then drawn to a capillary tube in a blast lamp. The tube and the sample are put in a beaker of test solution with capillary down, in a vacuum desiccator. The vacuum is applied and released, filling tube by suction to about  $\frac{1}{8}''$  depth over sample. The tube and the contents are then warmed to the required test temperature. The capillary is sealed and the tube is maintained at test temperature by keeping in an oven, or water or oil bath for the duration of the test which may be up to 3 months. At the end of this period, the solution is observed for colour, cloudiness or sediment and rated accordingly by reference to the relevant table. The sample is then taken out of the tube, wiped dry, and weighed immediately. It is observed for any visual disintegrative changes and rated.

The final volume of the sample is obtained by calculating quickly the specific gravity, preferably using a Jolly balance. From this data are obtained the changes in volume and weight which, on reference to the standard graphs, give the respective ratings. When all the 4 ratings are obtained, they are averaged to give one percentage rating. This gives the value of the sample under the test conditions. The test being not precise enough for a purely quantitative comparison, the results are also frequently reported as excellent, good, fair or poor.

These results are checked and calibrated against known results for standard chemical-resisting grades, in order that they can be used for prediction of field results. A very close concordance has been found to exist between the laboratory and the field results.

### Spectroscopic Identification of Timbers

A SPECTROSCOPIC METHOD FOR the identification of 16 varieties of Indian timbers is described (*Science & Engineering*, 1949, 2, 114). Aqueous extracts of the specimen (5 gm.) was prepared by allowing the pieces of timbers to remain in 100 c.c. of a 5 per cent solution of sodium hydroxide for 24 hr. Alcoholic extracts were obtained by extracting 5 gm. of the wood in 100 c.c. of an alcoholic caustic soda solution containing equal volumes of rectified spirit and caustic soda solution (0.91 gm./c.c.) for 24 hr. The filtered solutions were examined in a Hilger spectroscope of the constant deviation type fitted with a wavelength scale. A 100-watt pearl lamp was used for illumination.

TABLE I

TIMBER	AQUEOUS EXTRACT, WAVELENGTH ABSORBED	ALCOHOLIC EXTRACT, WAVELENGTH ABSORBED
	Å	Å
<i>Dysoxylum malabaricum</i>	4239	4250
<i>Terminalia bellerica</i>	4360	4405
<i>Calophyllum tomentosum</i>	4430	4270
<i>Lagerstroemia lanceolata</i>	4513	4275
<i>Tetrameles nudiflora</i>	4635	4350
<i>Xylia xylocarpa</i>	5770	4395
<i>Terminalia paniculata</i>	4790	4440
<i>Mangifera indica</i>	4742	4500
<i>Dichopsis elliptica</i>	5128	4600
<i>Cedrella toona</i>	5407	4710
<i>Terminalia arjuna</i>	4933	4850
<i>Tectona grandis</i>	5970	5205
<i>Albizia lebbek</i>	5334	5445
<i>Atrocarpus integrifolia</i>	5282	5670
<i>Atrocarpus hirsuta</i>	5384	5960
<i>Dalbergia latifolia</i>	5685	...

The position of the absorption bands of the extracts were recorded and mapped.

The difference in the absorption values observed for the aqueous and alcoholic extracts of a specimen serves as an additional aid in identifying a timber.

Table I gives the wavelengths absorbed by the different timber extracts.

The results of these studies show that ranges of absorption are specific for each wood extract and serve to identify a particular type of timber.

### New Sectioning Technique

THE APPLICATION OF THE ELECTRON microscope to many biological problems has been seriously hampered by the lack of a rapid practical method of cutting uniformly thin sections having adequate area and integrity of structure. Because of the very slight penetrating power of the beam in commercial electron microscopes and the great relative depth of field involved, specimen structure is difficult to interpret when sections are over a fraction of a micron in thickness.

The high-speed microtome has been of limited use in solving the problem. This precision instrument is not only expensive and complicated, but also produces a low percentage of useful sections. The recent method by Pease and Baker employs the standard histological technique of embedding the tissue in collodion and paraffin and a modified Spencer rotary microtome in which the unit of advance was reduced approximately to one tenth the calibrated value used to produce sections as thin as  $0.1\mu$ . This technique is not, however, handy because of the exacting demands made on the microtome-advancing mechanism and the difficulty involved in obtaining thin sections with standard embedding media.

A promising new procedure for obtaining extremely thin sections uses a methacrylic resin as an embedding medium, a thermal expansion device for advancing the specimen in a commercial microtome, and metallic shadow-casting for increasing observable detail in some of the sections. These techniques form the basis of the new method for producing thin sections suitable for obtaining transmission images at the higher magnifications in the conventional light, phase-contrast and electron microscopes. Polymerization of *n*-butyl methacry-

late provides a rapid and simple means for embedding the biological material in a solid resin. This gives an optically clear matrix from which the sections are cut, one at a time. Smooth, continuous advance of the embedded specimen toward the knife is effected by the thermal expansion of a brass specimen holder, which permits the microtome, with its advancing mechanism disengaged, to cut ultra-thin sections having uniform thickness, large area and integrity of tissue structure.

Before embedding, the tissues are fixed and then dehydrated by the usual cytological techniques. From absolute alcohol, they are transferred to a solution containing equal volumes of absolute alcohol and pure monomeric *n*-butyl methacrylate from which the inhibitor has been removed. After about 1 hr. in the alcohol-monomer mixture, the tissues are put in the monometer alone for an equal period. To ensure removal of the alcohol, they are then placed in two additional changes of monomer for at least 1 hr. in each.

No. 00 gelatin capsules are convenient embedding moulds. The main body of the capsule is set upright in a wooden block or other base and filled with the monomer, to which has been added 1 per cent (by weight) of a catalyst (2, 4-dichlorobenzoyl peroxide). After the tissue is placed in the mixture, the capsule lid is slipped on to retard evaporation, and the assembled capsule is placed in an oven kept at a temperature of  $45^{\circ}$ - $50^{\circ}\text{C}$ . For even heating, the capsules are suspended by strips of cellophane tape and good air circulation is maintained in the oven.

At the end of 6-8 hr., the monomer is polymerized into a solid matrix containing the tissue embedded at the bottom of the clear plastic. An additional period of several hours at this temperature will ensure complete cure. After soaking in water, the gelatin capsule may be peeled off the resin.

The device for holding and advancing the embedded specimen consists of a brass block with a hole threaded at one end to receive a standard  $\frac{1}{8}$ " brass pipe plug. A cavity drilled into the face of the plug provides a seat for the embedded specimen. Behind the plug is a needle valve, which admits compressed carbon dioxide. As the gas undergoes

a large change in volume, it cools and contracts the assembly. Stopping or reducing the flow of gas allows the apparatus to approach room temperature again and thus provides continuous advance of the embedded tissue toward the cutting edge.

In practice, the embedded specimen is first cemented into the mounting block with a mixture of pure gum-rubber and paraffin. Then, with the device clamped in the jaws of the microtome head, the entire assembly is cooled below room temperature. Upon the appearance of a thin layer of frost on the metal, the knife is adjusted so that the specimen just misses it on the cutting stroke. The specimen is then mechanically advanced at 2 or  $3\mu$  increments until the first slice is made. At this point the mechanical advancing mechanism of the rotary microtome is disengaged by setting it to zero and the gas flow is reduced or stopped. After a few seconds, the specimen can be cut again. Because the specimen is advancing continuously, a quick chopping stroke involving one complete revolution of the hand wheel is necessary. With a little experience, one can soon judge the necessary time interval between cuts. Some control of the rate of specimen advance can be obtained by bleeding the carbon dioxide at various reduced rates into the expansion chamber.

Although polybutyl methacrylate has excellent cutting properties, the sections usually are found to be somewhat folded. They are lifted from the knife with a dry camel-hair brush, picked up with a dissecting needle and placed on a water surface warmed on a hot plate to about  $35^{\circ}\text{C}$ . After a period ranging from a few minutes to an hour or more, many will flatten out on the surface and exhibit bright interference colours. These sections are then floated on to clean microscope slides and allowed to dry flat. Sections prepared for phase contrast microscopy are placed in acetone or toluene for about  $\frac{1}{2}$  hr. to remove the matrix and are mounted in Canada balsam. For ordinary light microscopy, the matrix is dissolved off and the section stained in the usual way.

In preparing material for the electron microscope, the sections are floated from the water on to clean glass slides and dried flat. The matrix is then dissolved out by placing the slide in acetone, toluene, or amyl acetate.

A dilute solution of collodion in amyl acetate is allowed to flow over the slide bearing the tissue, which is then permitted to dry at room temperature. The collodion film containing the section is floated from the slide on to water, and the specimen-mounting screens of the electron microscope are placed over the area of the film containing the section in the usual manner.

This process of wide application in electron microscopy produces a three-dimensional aspect as well as greater contrast in structural details of the tissue.

Although this new method for obtaining very thin sections has given satisfactory results, it possesses certain limitations. Knife sharpness, for example, is of vital importance. The tilt of the knife during sectioning is not particularly critical. The greatest chance for failure appears to lie in the polymerization of the embedding mass. Use of low temperature catalyst and maintenance of a curing temperature of 45°-50°C. will usually prevent the formation of insoluble resins. Occasionally, tissues are injured during the polymerization reaction and such tissues, which are easily detected, can be promptly discarded. For this reason, the use of fixing solutions with good hardening properties is recommended. While fixation artefacts remain problems for serious consideration, the new technique provides an inexpensive, practical method for producing ultra-thin sections of tissue in almost a routine fashion (*Science*, 1949, **110**, 66).

### Recent Advances in Instrumentation

A WIDE VARIETY OF NEW LABORATORY instruments has been developed and announced in the U.S.A. during recent months. These range from new instruments to detect atomic energy radiations to a new vacuum-tube voltmeter for measuring voltage in electronic and other low-energy circuits (*Chem. Age*, 1949, **61**, 155).

**Resistance Thermometer**—A new thimble-size platinum resistance thermometer using the same temperature-sensitive element as the standard platinum thermometer but  $\frac{1}{4}$ th its size, measures temperatures in the region of 10°K and higher in calorimetric experiments. The instrument can also be employed

as a primary standard for resistance thermometry between -190° C. and +500°C. With the 2-G Mueller bridge temperature, measurements can be made to an accuracy of  $\pm 0.01^\circ\text{C}$ .

The thermometer has a platinum protecting tube, about 48 mm. long and 5.6 mm. outside dia. and can be readily mounted entirely inside a calorimeter by casting in low-melting alloy. Being helium-filled, it is usable down to 5°K. Four-wire current and potential leads are brought out through a glass seal. Nominal resistance at 0°C. is 25.5 ohms.

**Potentiometer**—The K-2 potentiometer of *Leads & Northrup Company* combines precision with facility of operation so essential for studying tiny high-speed particles constituting beta rays. Its chief features are the addition of a third and lower range, elimination of exposed electric conducting parts and standardization on any of its 3 ranges (0.0-0.161, 0-0.161 and 0-1.61). The last  $\frac{1}{10}$ th,  $\frac{1}{100}$ th or  $\frac{1}{1000}$ th volt is spread over a 5 meter slide-wire, making it easy to follow a small fluctuating voltage with accuracy not ordinarily possible where several switches must be manipulated within the range of fluctuations.

The potentiometer can be used for various measurements of potential difference, hydrogen-ion concentration, oxidation reduction, polarization, temperature, and other measurements.

**Consistometer**—A new apparatus, the "consistometer", with a simple design and high efficiency, for measuring the flow characteristics of lubricating greases at constant temperature as the grease is being mechanically worked has been devised. It can similarly determine the apparent viscosity of rubber solutions at different shear and temperature. The principal advantages of the apparatus are its flexibility, wide range of shear and consistency measurements, short test cycle and provision for characteristics flow data while working under controlled conditions.

The consistometer is composed essentially of two coaxial steel cylinders mounted vertically, with a capillary-type shearing element in between through which grease under test is forced in either direction by close-fitting pistons. Dead weights on the down-stroke and air pressure on the up-stroke are used on the pistons while making flow measurements. Provi-

sion is also made for automatic mechanical working. Possible rates of shear cover a rather wide range, from about 10 to 100,000 reciprocal seconds.

**Calibrating Hygrometers**—A new humidity test apparatus for research, calibration and testing of hygrometers and various humidity instruments at low temperatures has been developed at the U.S. National Bureau of Standards.

A current of dry air is divided into 2 streams (by a proportionating valve) one of which is maintained dry, while the other is completely saturated over a series of trays containing ice. The 2 currents are then mixed in a mixing chamber and allowed to escape through a test chamber into the atmosphere. All the 3 chambers—the saturator, mixing chamber, and test chamber—are kept immersed in a constant temperature bath. Essential functional units of the apparatus are: the drying system, the proportionating system, the humidifying system, the test chamber, the cooling system, and the thermoregulating system for temperature control.

The instrument fills a basic need in meteorology an is expected to find application in refrigeration, air-conditioning and other fields in which humidity plays an important role.

**Freezing Point Determination Apparatus**—A precision apparatus involving a platinum resistance thermometer and bridge for evaluating purity of hydrocarbons by measuring their freezing points has been developed by the U.S. Bureau of Standards. Procedure employed in the determination utilizes time-temperature observations of the liquid solid equilibrium in freezing and melting experiments.

5 c.c. of the sample to be examined are placed in an inner vacuum flask surrounded by a refrigerant and stirred by an overhead electric motor. Observations of time and temperature are made, starting with the temperature of the liquid above the freezing point and continuing until stirring becomes difficult. By plotting these values, using a suitable scale on the temperature axis, it is possible to determine graphically the "zero time" at which crystallization would have begun in the absence of undercooling. The corresponding temperature value is then the true freezing point. The purity is

calculated from the measured freezing point as well as the value of the freezing point for zero impurity and the cryoscopic constants.

Determination of freezing points for substances such as ethylbenzene, which do not normally yield time-temperature freezing curves, is made from melting curves. The procedure is practically the same as for freezing experiments except that when stirring becomes difficult, the cooling bath is replaced by an appropriate warming bath.

**Air Meter**—Development of "Anemotherm", a three-way air meter providing air velocity, air temperature and static pressure readings at the turn of a knob is reported. The meter is expected to find wide application in adjusting and testing equipment used for heating, ventilating and air-conditioning.

The operation of the instrument on self-contained batteries and provision of the small probe attached to a long flexible cable make the instrument portable and permit readings even in the neck of the air diffuser. The meter measures air velocity from 10' per min. to 500' per min., provides air-response measurement of temperatures from 30°F. to 155°F. Static pressure, either negative or positive, may be read directly in in. of water from 0.05 to 10 positive, and 0.05 to 4 negative. The new instrument detects even the slightest draughts and measures air velocity accurately regardless of direction of air flow.

**Raman Spectrograph**—This instrument, designed by the *Esso Laboratories*, is based on the discoveries first made by Sir C. V. Raman in 1928.

The photo-electric recording spectrograph, which is capable of measuring the amount of light from a candle 5 miles distant, has found application in the analysis of petroleum products in the laboratories of the *Standard Oil Development Co.*

### Measurement of Thickness of Microtome Sections

A SIMPLE AND ACCURATE MEANS for measuring the thickness of very thin microtome sections has been described (*Science*, 1949, 110, 73).

Several optical methods are available for measuring thin films but their use is difficult when the area of the section is of the order of a sq. mm. A much

simpler way is to measure, under the microscope, the area of the section, then to melt the section into a sphere from whose diameter the volume of wax can be calculated. To obtain the sphere, the section is caught on a very thin (less than 10 $\mu$ ) glass fibre, and the fibre is mounted in the field of a microscope. A small loop of electrically heated wire is carefully guided close to the section, which is slowly melted. If the glass fibre is thin enough, an almost perfect single sphere will result; for greater accuracy, one may prefer to calculate its volume as an ellipsoid. It is important to watch the heating under fairly high magnification, and to heat slowly, as too high a temperature will cause evaporation.

### Estimation of Fatty & Rosin Acids

A SWEDISH METHOD FOR accurate estimation of fatty and rosin acids by complete esterification of the fatty acids is described (*Chem. Age*, 1949, 61, 184).

The mixture is esterified with *n*-butanol and benzene and the water formed is separated. Complete esterification of the fatty portion takes place but at the same time rosin acids are also partially esterified. To reduce this, as far as possible, a suitable catalyst (benzene sulphonic acid) is used in low concentration, which is constant, and the temperature of the reaction is kept down during treatment. The concentration of the catalyst being constant, a definite amount of rosin acids is esterified per time unit. Thus, it is possible to introduce an exact correction for the fraction of rosin acids converted during cooking.

The unesterified rosin acids in the reaction, mixture are determined titrimetrically. If the acids are of varying molecular weights, gravimetric methods have to be applied.

### Palm Oil Substitute

A SYNTHETIC SUBSTITUTE FOR THE expensive African and East Indian palm oil, which is extensively used in the manufacture of hot-dip tin-plate, has been developed at the *Armour Research Foundation*, Chicago (*Chem. Age*, 1949, 61, 185).

In making hot-dipped tin-plate, sheets of pickled steel are passed through a flux into a bath of molten tin and through an 18"

layer of hot (450°F.) palm oil. The palm oil substitute has therefore, to be one which makes smooth, bright and quality tin-plate, drains rapidly and freely from the sheet, neither oxidizes nor polymerizes readily and has low viscosity at 100°-200°F. so that it may be pumped or poured readily. Moreover, the oil has to be non-toxic, odour-free and preferably edible; should not become rancid during storage, or interfere with lithographing, lacquering or soldering procedures and should retard atmospheric corrosion.

It has been found that dimerized linoleic acid (dimer acid) consisting of 2 linked molecules of linoleic acid has the best possibilities. Its flash point (600°F.) is even higher than that of palm oil (470°F.) and its volatility is one-third of the African product. A mill test on this material was encouraging except that the acid tended to increase in viscosity.

### A New Industrial Amine

A PRIMARY AMINE—ROSIN AMINE D, of great industrial utility—has been developed by the *Hercules Powder Co.* Derived from modified rosin, the amine is a pale-yellow, viscous liquid having a density of 0.997 at 25°C. Commercial qualities have a purity of 88-92 per cent. The amine is soluble in most organic solvents but is practically insoluble in water. With most of the film formers and resins, it is compatible. At room temperature, it forms salts with mineral acids and organic acids of low molecular weight. With high molecular-weight organic acids, the reaction takes place at 70°-95°C. Carbon dioxide combines with the amine to form a white crystalline unstable salt. Within a specific range of concentrations of 0.02-0.4 per cent, the salts of the low molecular-weight organic acids exhibit hazy or cloudy solutions with distilled water.

Inorganic salts of Rosin Amine D are practically insoluble in water or organic solvents. The pH values of their dispersions and of dilute solutions of soluble salts range from 5.4 to 6.8, the values decreasing with higher concentrations. The amine and its carboxylic acid salts give a positive Libermann-Storch test for rosin.

Non-ionic type emulsifiers such as Tweens and Renex can emulsify the amine and its salts in water



solutions; ionic type emulsifiers, e.g. sulphonated castor oil, Naccanocol and rosin soaps are not suited on account of their reaction with the amine to form salts. Fatty acid soaps, if used with concentrated amine solutions, cause gel formations.

Rosin Amine D and its salts are stable up to 100°C. They darken slightly on ageing in air at normal temperatures. On heating at 100°C., slight decomposition with loss of nitrogen takes place, and higher temperatures induce further darkening and decomposition.

The amine does not cause any appreciable corrosion of metals. It is non-toxic; concentrated forms irritate the skin slightly due to its weak alkaline action.

The chemical is used as a flotation agent, rubber softener, corrosion inhibitor, herbicide in the control of crab grass, preservative and germicide, and as a chemical intermediate in the preparation of numerous derivatives. Its addition to asphalt, mastic cements improves adhesion. In ceramic inks its use gives better flow and spreading properties. The amine and its derivatives are expected to find wide application in breaking oil emulsions in the production of crude petroleum.

### Synthetic Detergents

THE PRODUCTION AND THE USES of synthetic detergents in the United States are described (*C.T.J.*, 1949, 125, 33).

The first commercial production of synthetic detergents was in Germany during the early 1930's, when the extreme shortage of fats and oils led to the development of a series of sulphonated alcohols which had superior detergent properties. The better types of synthetic detergents possess the following general properties: (1) excellent wetting ability; (2) neutrality in solution (non-hydrolysing); (3) solubility and effectiveness in cold water; (4) ready rinsability; (5) resistance to hard and sea water and acid and alkaline solutions; (6) excellent foaming properties; and (7) tastelessness.

One important technical weakness was recognized early for most synthetic detergents, that is, that they are not equal to soap for the removal of heavy oils, particularly from cotton fabrics. Whether a synthetic can compete with soap in soft water as well as hard water depends on the following considerations: (1) quality and price

believed to be satisfactory; (2) ability to wash heavily soiled cottons: not yet comparable with low-cost detergents, but potentially so; (3) capability of being made into bars: not yet satisfactory; (4) supplies of chemical raw materials: capable of eventual solution.

Listed below are the chief types of synthetics falling in the anionic and non-ionic groups:

**Anionic** — (1) Alkyl aryl sulphonates; (2) sulphated fatty alcohols; (3) miscellaneous sulphates and sulphonates: (a) alkyl sulphonates; (b) sulphated esters and acids; (c) amide sulphates and sulphonates; (d) sulphated and sulphonated oils, fats and waxes.

**Non-ionic** — (1) Fatty esters of glycerol and glycol and other polyhydric alcohols, such as sorbitol; (2) condensation products of ethylene oxide with fatty acids, alkyl phenols and mercaptans.

Within the types of compounds mentioned, there are literally hundreds of possibilities.

The alkyl aryl sulphonates, because of their excellent properties and low price, have been pushed as a competitor for soap in household cleaners, especially in hard-water areas. In addition they have wide industrial usage.

Sodium lauryl sulphate represents the bulk of the sulphated fatty alcohol detergents, and is prepared from coconut oil. At present the bulk of this type of product is marketed as household powders.

There has been considerable interest in the past few years in the non-ionic synthetic detergents because of their good detergency and low foam. The principal drawback to these materials are their inherent high price and liquid form.

With the use of "builders", sodium tripolyphosphate and tetrasodium phosphate, it is possible to produce an ideal household detergent using synthetics.

### Prevention of Corrosion in Metals

RECENT METHODS FOR PREVENTING metal corrosion were reviewed at the recent U.N. Scientific Conference on the Conservation and Utilization of Resources (UNSCCOUR). Humidity control, de-aeration inhibitors, cathodic protection and alloying materials were some of the methods discussed.

**Humidity** — At relative humidity below 30 per cent, corrosion is negligible. Air-conditioning in

large spaces and suitable moisture absorbing substances such as silica gel with packaged apparatus can accomplish this. When the package is a metal container, the inside air can be replaced with an inert gas such as nitrogen.

**De-aeration** — Removal of atmospheric or other oxygen from the environment, e.g. the use of a de-aerator in the treatment of water for boiler-feed and steel pipe lines inhibits corrosion.

**Inhibitors** — Certain compounds which function mostly as chemically or physically absorbed films either alter the electro-chemical characteristics of the metal or serve as mechanical barriers to the normal corrosion processes. Reactions of various chemical inhibitors are described.

**Cathodic protection** provides the most effective means of arresting corrosion. It is based on the principle that most corrosion in practice is electro-chemical in nature and results from the flow of current through an electrolyte between areas of different potential which may exist on the surface of the single metal or between two or more different metals. The solution is to bring all surfaces to the same potential. This is done by introducing a current to offset the one produced by the difference in potential, i.e. by discharging current on the more cathodic surfaces, so as to achieve their cathodic polarization to the potential of the adjacent anodes. The method finds the most important application in connection with miles of underground oil, gas and water pipe lines and power and communication cables (*Sci. Newsletter*, 1949, 56, 4).

### Liquid Oxygen Converter

A NEW AUTOMATIC AND COMPACT liquid oxygen converter for aviation, industrial and medical purposes has been developed at the U.S. National Bureau of Standards (*Chem. Age*, 1949, 61, 255).

The equipment, 25" high and 18" dia., weighs 60 lb. and holds 62 lb. oxygen enough for 10 men for 10 hr. It is sturdy, simple in design and operation, attains pressure rapidly, consumes less oxygen and delivers warm gas. It consists of a standard 25-litre metal Dewar flask modified by the addition of a bottom drain, together with 2 coils, one for build-up of pressure and the other for warming the gas as it is delivered. After it is filled with liquid oxygen, the flask can be sealed off from the atmosphere.



To build up pressure, a valve is opened to allow liquid to flow through a drain tube and liquid trap at the bottom of the flask and into the build-up coil where it is evaporated and warmed by the atmosphere. The warm gas rises through the coil by thermal convection until it enters the top of the flask where it mixes with the gas above the liquid. Here the gas that condenses on the liquid furnace is immediately replaced by freshly warmed gas and the cycle continues, the gas pressure rising quickly. The fact that it is not necessary to heat the entire mass of liquid to raise the pressure constitutes an important difference between this and earlier types of liquid oxygen converters.

The equipment delivers gas at a temperature differing by only about 5°C. from the atmospheric temperature for flows up to 150 litres per min. When the container is full, pressures from 0 to 65 lb. p.s.i. are built in 10 sec. and are quite stable. The equipment has successfully withstood severe heat, cold and vibration tests. Operation is reported to be satisfactory at altitudes up to 40,000'.

### Black Stainless Steel

A NEW TYPE OF STAINLESS STEEL, the "shineless stainless steel", is finding application for many industrial purposes. Like the conventional bright stainless steel, this black variety has a thin chromium oxide coating and the blackening of the surface is achieved by placing the metal in a molten bath of dichromate at a temperature of 730°-750°F. for 15-30 min. The black surface, which is resistant to the same degree as the ordinary stainless steel because of its non-reflecting surface is finding application in military requirements. It is also used in certain types of jewellery (*Sci. Newsletter*, 1949, 55, 394).

### New Uses for Rubber

A NEW INDUSTRY FOR MANUFACTURING rubber hydrochloride from natural rubber and hydrochloric acid is being developed in Holland. A simpler and far more economical process than the American for the manufacture of rubber hydrochloride (Pliofilm) has been perfected. The process will enable natural rubber to compete with synthetic rubber and plastics.

Rubber hydrochloride has been found very suitable for packing

purposes especially where water-clear transparency and very low moisture permeability are required. Another important use of natural rubber announced is the manufacture of ebonite tanks for storage of solid and liquid corrosive chemicals. These tanks are strong, stable and inexpensive (*Chem. Age*, 1949, 61, 122).

### Fire Resistance & Sound Insulation of Walls

THE STRUCTURAL STABILITY, FIRE resistance and sound insulation requirements of "party walls" between houses are dealt with in the *National Building Studies Special Report No. 5* published by the *Department of Scientific & Industrial Research*, U.K. (*H.M.S.O.*, London, Price 2s.). The report discusses the twofold problem: firstly to determine standards of performance in respect of fire resistance, and secondly to find ways of assessing whether particular forms of construction will attain the requisite standards.

Until the middle of the 19th century, the need for fire protection was the major consideration in the design of "party walls" apart from the obvious requirements of structural stability. A solid wall of 9" of brickwork was the accepted and adequate form of construction. Since then the noise problem has so increased as to render this wall inadequate as a barrier to noise, while new methods of construction introduced in recent years are impracticable because of their high cost.

The design of the wall should be such as to meet the following specification: A party wall should provide a complete separation between houses, from foundation level to the underside of the roof covering. It should afford at least 1 hr. fire resistance and should provide an average sound reduction of 55 db. between the living rooms in one house and the living or bed-rooms in another house; and 45 db. between other rooms.

Higher grades of fire resistance should be provided between houses of combustible construction and at defined intervals in terrace houses. In the latter case, the wall should, in addition, be self-stable.

Of the single and multiple-leaf systems of construction, the four-leaf system in which the individual leaves are insulated and have a total weight in leaves of 12-15 lb./sq. ft., appears to give an average sound reduction of 55 db.

To avoid reduction of fire resistance by perforation of the wall with floor joists, the joists should preferably run parallel with the party wall. This is also a useful measure in respect of sound insulation.

Indirect sound transmission due to common floor beams and wall plates can be overcome by using a double-frame system of construction which can be more easily adapted to give the necessary standards both of fire resistance and sound installation than single-frame systems.

Weight for weight, such materials as clinker concrete, foamed slag and wood-wool appear, on present evidence, to be better sound insulators than the denser materials such as dense concrete or brickwork.

### Indian Pulp & Paper

THE ANNIVERSARY NUMBER of this journal (July 1949; price Rs. 3), in addition to the usual features, contains some excellent and instructive articles. Articles of interest include: Research for the Paper Industry; Nature of Bamboo Lignin; Paper; Origin, History and Manufacture; Photography as an Aid to the Paper Mill Chemist; Water Marks, etc.

### Electronic Measuring

THE *Philips Laboratories*, EINDHOVEN, Holland, have issued 9 numbers of the second volume of their technical bulletin entitled *Electronic Measuring* in which are discussed the applications of electronic measuring technique. The bulletin gives an idea of the significance of new techniques and developments in electronic measurements. The publication is supplied free of charge on request to *Philips Electrical Co. (India), Ltd.*, "Philips House", 2 Heysham Road, Calcutta 20.

### REPORTS FROM STATES & PROVINCES

(Continued from page 463)

160,000 seedlings to plant about 2,000 acres. Seednuts from selected trees having all the desirable characters will be collected mainly in the months of February to June and the supply of seedlings will commence in July and continue throughout the monsoon months.

# Reports from States & Provinces

## MYSORE

### Sugar Factory for Shimoga

MYSORE STATE, ONE OF THE MAIN sugar-producing areas, will have another sugar factory situated at Shimoga, in the Malnad area. The factory is estimated to cost Rs. 50 lakhs on capital expenditure and Rs. 15 lakhs as annual recurring expenditure. The necessary licence for obtaining the plant and machinery has already been obtained.

### Fertilizer Factory

CONSTRUCTION WORK ON THE Government chemical and fertilizer project estimated to cost Rs. 3 crores and capable of producing 25,000 tons of ammonium sulphate and 25,000 tons of nitro-chalk annually is to commence by the end of this year. The necessary import licence for the plant has been obtained and plans are being finalized in consultation with the *Chemical Construction Corp. Ltd.* of New York.

## RAJASTHAN

### Beryl Resources in Ajmer-Merwara

THE ANNUAL OUTPUT OF BERYL from the mica pegmatites in Bihar and Madras would probably be 5-10 tons during mica mining. The principal source will be Ajmer-Merwara, where the mineral occurs more extensively than in other parts of the Indian Union; these sources are said to have supplied about 40 tons a month during the war years.

Further detailed mapping in parts of Assam, Orissa, Bihar and Central Provinces, Hyderabad and Madras, may expose some beryl-bearing pegmatites. On a conservative estimate a few hundred tons per annum may be raised in the Indian Union.

In Ajmer tehsil, the most important beryl workings are located near Lohagal and Makrera, the latter with a total production between 500 and 1,000 tons. Promising localities are

Foya Sagar Lake, Qazipura, Boraj, Barla and Rajosi. Indications are noticed in the neighbourhood of Chamunda, Palran and other localities. In Beawar region, the localities from where large quantities of beryl were hitherto obtained are within 3-4 miles from Jawaja. Dhoti Danti in Kharwar is a noticeable locality. In Keki area the important localities are Bogla, Gulgaon, Para, Gonda, Kacharia, Kalera, Khawas, Khoda, Nemode and Bisundini. The total production from Bisundini mine is known to have varied from 500 to 1,000 tons. The promising occurrences in Nasirabad area are Tihari, Tilara, Tantuti, Shokla, Loharwada, Hanwantia and Kanpura. The deposits in Ajmer-Merwara are by no means exhausted and large quantities of beryl are yet to be forthcoming under favourable conditions of mining industry. On an average, about 50-100 tons per annum may be easily obtained from Ajmer-Merwara.

The systematic survey of the beryl occurrences in Ajmer-Merwara has shown that usually large workable deposits of beryl are not present with good quality ruby mica. Large-scale mining for low grade mica, may yield large quantities of beryl as by-product.

## MADRAS

### Breeding of Improved Varieties of Millets

*Rust Resistant Strain of Panicum italicum* (Korra)—This millet crop, common in the ceded districts, is invariably susceptible to the attack of a rust, characterized by rusty, brown spots on the leaves. This rust disease is responsible for poor yields. A selected strain, S.L. 3756, evolved at the Millet Breeding Station, Coimbatore, has been found to be comparatively resistant to the rust and yields a bigger harvest.

*Improved Strain of Irrigated Sorghum vulgare* (Cholam)—A high-yielding strain, K. 2, has been found suitable for cultiva-

tion in two seasons—January/February and April/May—in certain districts. The strain matures earlier than the local varieties. The cultivation of the strain not only saves the cost of one irrigation but also gives extra produce valued at Rs. 37-8-0 per acre at the present price for the millet.

*New Eleusine Corocana* (ragi) strain—K. 1, a ragi strain, yielding 18 per cent more than the local variety (288 lb. per acre) isolated at the Agricultural Research Station, Koilpatti, is now available for distribution. A net profit of Rs. 36/- per acre is expected out of its cultivation.

*Hybrid Varieties of Pennisetum typhoides* (Cumbu) Two new hybrid varieties of *cumbu*, X. 1 and X. 2, have been recently released for trial from the Millet Breeding Station, Coimbatore. They have been produced by crossing promising pure lines which exhibited the maximum hybrid vigour when crossed. Trials conducted in Tiruchirapalli district have been encouraging.

*Green Manure*—An acre of paddy field requires 25-30 lb. of small-sized green manure seed like *Pillipesara*, *Sesbania speciosa*, etc. Efforts to produce seed of this green manure crop on the paddy-field bunds at the several agricultural research stations show that it is easily possible to obtain 25 lb. of seed from *Sesbania speciosa* planted on the well-trimmed field bunds of an acre of land immediately after planting paddy in July-August. Planting of seedlings is to be preferred to dibbling seed directly on the bunds. Nursery of *Sesbania speciosa* should be sown on a small high-level plot 4-5 weeks in advance of the completion of paddy planting.

### Coconut Seedlings

IN ORDER TO SUPPLY THE PUBLIC with selected seedlings at comparatively low price, a comprehensive coconut nursery scheme financed by the Government and the *Indian Central Coconut Committee* was sanctioned by the Government of Madras in October 1948, and was put into operation on 10th November 1948. Nurseries have been started at the 8 research stations, viz. Anakapalle, Samalkot, Marutera, Tindivanam, Pattukottai, Coimbatore, Pattambi and Nileshtar. Under this scheme, it is proposed to produce annually

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# INDIAN PATENTS

The following is a list of Patent Applications notified as accepted in the *Gazette of India*, Part II, Section 1, for September 1949. The patents from the *Council of Scientific & Industrial Research* are indicated by asterisks.

## Paints, Varnishes & Lacquers

- \*40752. SIDDQUI, SARIN & SINGH : Conversion of bhillawan shell liquid to a non-vesicating drying product for the manufacture of lacquer varnishes, stoving enamels, water resistant and flame resistant paints : *Treating bhillawan shell liquid with chlorine until chlorine content is at least 25-30 per cent.*

## Organic Chemicals

41029. UNITED STATES RUBBER COMPANY : Process of making DDT (22nd July 1948) : *Characterized in using fluosulphonic acid as condensing agent in a continuous process.*

## Miscellaneous Chemicals

40983. SOLVAY & CIE : Process for oxidizing substances with chlorite solutions : *The substance is immersed in a suspension of activating agent and then it is dipped in the chlorite bath.*  
40984. SOLVAY & CIE : Process of activating chlorite solutions and bleaching materials with these solutions : *Introducing into the chlorite solution a hydrolysable organic solvent.*

## Instruments (Professional, Scientific & Controlling)

41665. ROY : An improved balance : *Balance provided with ball bearings at one or more points of movement.*

## Food & Kindred Products

39650. DIJK : Improved method, process and means for decorticating cashew nuts and like products : *Freezing and cracking the nuts and then separating shell from kernel.*  
40835. PITHWALA & SREENIVASAN : Preparation of a modified tamarind seed polyose suitable for food industries : *Tamarind seed polyose is subjected to the action of citric acid.*  
39384. THE DOOR Co. : Improvements in clarification of sugarcane juice : *Passing lime into admixture with mud in the lowermost compartment of a clarifier.*  
40949. ANHEUSER-BUSCH, INCORP. : A process for producing riboflavin, yeast or vitamins of the B complex : *Adding a culture to the nutrient medium, containing the culturing and separating the cellular material.*

## Drugs & Pharmaceuticals

38838. N. V. PHILIPS' GLOEILAMPENFABRIEKEN : Methods of preparing medicinal vitamin C

tablets : *Mixing 10 gm. of vitamin C per gram of a mixture of cocoabutter, sugar and cocoa.*

41357. GLAXO LABORATORIES LTD. : Preparation of liver extracts : *Passing the aqueous extract of liver through a silica gel column and thereafter eluting the active principle.*  
41358. GLAXO LABORATORIES LTD. : Preparation of liver extracts : *Subjecting an extract of liver to a counter current extraction process between water and a suitable solvent as defined herein.*  
40472. CHAUDARY : Improved driving arrangement for cycles : *Another free wheel on opposite side of rear wheel axle, carrying a length of chain.*

## Metal & Metal Products

40929. DESAI : Extraction of metal copper from malachite or ores containing copper carbonates : *Reacting ferrous sulphate of 5N strength with malachite to form copper sulphate to which scrap iron is added.*

## Rubber & Rubber Products

41067. SINGH : An improved process of manufacturing rubber balloons : *Liquid latex is allowed to fall from the orifice of a container into a rotating former.*

## Textile & Textile Products

41119. INDIAN JUTE MILLS' ASSOCIATION RESEARCH INSTITUTE : Preparation of a fast-to-light substantially white jute (Addition to No. 37868) : *Treating bleached jute with acetic anhydride at a temperature below its boiling point in the presence of an alkali metal acetate.*

## Miscellaneous

39131. DEWAR & MCLEOD & Co. LTD. : Tea-breaking machines : *Breaking effected by pressure and bending applied to the belt and to tea on the belt.*  
9660. CHAUDARY : An improved plough : *Two tilling points spaced apart from one another and at different levels.*  
\*40112. NARAYANA RAO & SREENIVASAYA : A process for production of distillery yeasts of high potency : *Securing a highly active zymase complex within it and elaborating protective agents within the cell, which stabilize the zymase complex.*  
40600. SINGH : Typewriters for Hindi, Devnagri and like scripts : *Half characters provided on type bodies and vertical stroke provided on a linear adjacent to guide, typebar actuating the lever when half character key is depressed.*

## Science & Export Promotion

MUCH attention has of late been given to the question of stimulating India's exports in the interest of balanced trade. The Export Promotion Committee (Chairman, Sri A. D. Gorwala), appointed last July, has made valuable recommendations in this regard, some of which have been accepted by the Government and others are receiving consideration. It is axiomatic that in the long run the exports must pay for imports if the country's economy is to be based on sound foundations. The need for stimulating export trade to enable the financing of imports without frittering away the valuable sterling resources is, therefore, obvious.

The Export Promotion Committee has carried out a searching analysis of the impediments in the way of export trade, the most serious among them being the maladjustments in the internal economy resulting in high prices. Due to the upward trend of Indian costs "some of our goods price themselves out of all markets". Speculative activity has been largely responsible for the high prices. Export restrictions, absence of standardization, unethical practices of some of the exporters, sales tax barriers, income tax difficulties, transport shortage, to mention only a few, are among the contributory causes that have hampered export expansion.

The Government has accepted the recommendation of the Committee that special steps should be taken to develop and promote Indian export trade. An export drive, however, can be successful only if the volume of internal production expands. The production of exportable cash crops is to be encouraged, at the same time securing adequate production of food crops. Export control is to be further liberalized, particularly with regard to manufactured goods. Processing and re-export of goods of soft currency origin are to be encouraged, and

steps are to be taken to improve the quality of Indian exports to obviate frequent complaints from overseas buyers on this score.

These and other measures, important as they are, for easing export difficulties and so stimulating exports, have to be supplemented by others, to some of which the Committee has drawn attention. Among them is the setting up of an Export Promotion Department to co-ordinate the various aspects of export policy, to collect and disseminate knowledge on export commodities, to watch and supervise the progress of exports, provide guidance to exporters, and to take all measures necessary for the spread of Indian trade by publicity in all its forms without and within the country. For the collection of information from abroad, the main source will be the Trade Commissioners in various countries, whose principal function is clearly the nursing and expansion of export trade. Market research into tastes is not considered so important at the present stage, and surveys by agencies primarily concerned with exporting special articles, supplemented by a continuous flow of knowledge through Trade Commissioners should prove adequate.

India's exportable commodities form an impressive list, and include such items as jute manufactures, tea, cotton piecegoods, raw jute, oils and oil seeds, raw cotton, hides and skins, mica, shellac, sugar, coal, spices, manganese and other ores, cottage industry products, fruits, nuts and vegetables, horns and hoofs, crushed bones, tyres and tubes, manufactures of wood and timber, fish, pharmaceutical products, tobacco, chillies, magnesium chloride, vegetable products (*vanaspathi*), myrobalans, carpets, raw wood, coir matting, aluminium utensils and tanned hides. A list of this type is of a fluctuating character. Commodities like indigo and madder, which were once prominent among exportable commodities, no longer figure in it. The position of a few others is threatened

with synthetic rivals. The export position of a good many is capable of being strengthened by improvements in quality, standardization, by processing them into finished products before export, or by other means. New items of exports can be searched out and added to the list from among India's variegated natural resources.

The Export Promotion Committee, faced with the problems of the present, have not discussed in detail one essential prerequisite for any long-term planning of export promotion. We refer to the rôle played by scientific research in stimulating export trade. History of industrial development since the time of the industrial revolution is replete with instances which show that science has influenced the trend and pace of trade and commerce. Trade has its problems; and every year these increase instead of becoming less. New industrial wants come to light and have to be met. New materials have to be produced. The raw materials of industry, provided by Nature, are highly complex, and a full understanding of their fundamental nature, properties and reactions are essential for their optimum utilization in trade and commerce — an understanding revealed only through scientific investigation and research.

A proper scientific assessment of India's raw materials and their potentialities is a desideratum for any long-term programme of export. In this connection mention may be made of such an attempt by the *Council of Scientific & Industrial Research* in compiling the *Dictionary of Economic Products & Industrial Resources of India*. This work would constitute a useful guide not only to the administrator concerned with the study of exportable products but also to the trader himself. Further, it is high time that the Government of India brings out a publication listing in brief the various available materials, their potentialities for export, and the controls to which they are subject. This publication would be on lines of the *Raw Materials Guide* published periodically by the *Board of Trade* in the United Kingdom.

The value of resources for export can be considerably enhanced through the application of science. Reference has been made to standardization and quality control in the preceding paragraphs. It should be remembered that failure to keep up standards, even in a few cases, is likely to affect the reputation of Indian goods as a whole, and injure the country's trade. Instances of complaints

from consumers regarding sub-standard products are numerous, and any one engaged in listing India's exportable commodities cannot but feel amazed at the large number of them subject to gross and wilful adulteration. Modern industry, involving mechanized processes, demands raw materials of invariable quality, and consumers, unless assured of steady supplies of standard materials, are forced to seek substitutes, preferably factory made, of proved and dependable quality.

The threat to natural products from synthetics is ever present, and it behoves all producers of primary products to remember the fate of the natural indigo industry and profit by history. Japan met the situation created by synthetic camphor which threatened to ruin her natural camphor industry by invoking the aid of science not only to improve the efficiency of production but also to build up a formidable consuming industry, and improved her position as an exporter of camphor and camphor products. Among other industries based on scientific research which have secured for Japan an invincible position in the export trade are the sericulture industry and the artificial pearl industry. Science can, therefore, help in improving the raw materials of industry and strengthen export trade. Jute is now India's best dollar earner, and scientific research should be directed towards increasing the yield and quality of jute. Recent reports from Calcutta indicate the great possibilities for jute improvement by X-ray irradiation. Any promising approach to improvement, such as to the one indicated by X-ray irradiation, should be pursued with vigour to its logical conclusion, and more and better jute should be produced. Similar investigation with other economic products, cotton and oil seeds for instance, are equally important from the export point of view. The production of oil seeds with higher oil contents or better size characters would enhance their export possibilities. Similarly, barley with improved malting qualities would provide an expanding export market for Indian barley, the demand for which has now greatly diminished.

The export value of a material is enhanced through processing it into a finished or semi-finished product and exporting the treated material. An instance in point is raw hides vs. tanned leather. There is no reason why, through the application of knowledge revealed by scientific research, raw hides should not

It is manifest from this cursory survey that scientific research can help not only in

assuring and improving the export value of Indian products, but also in revealing new possibilities for export. The processes which the trained research mind employs in tackling a problem, namely "gets history of problem ; surveys literature ; defines problem ; studies attack ; suggests solution ; determines needed data ; selects methods ; outlines programmes ; tests solvability ; verifies findings ; collects data ; systematizes data ; analyses data ; appraises results, makes deductions ; reaches conclusions ; tests conclusions ; and plans developments ", provides a guarantee that a clearly stated problem — export promotion of Indian products, in this case—can be tackled satisfactorily through the help of science. Strengthen science, you strengthen export possibilities. Research establishments in this country, in co-operation with chambers of commerce, can provide answers to many problems facing trade and commerce.

S. S. BARAL, R. K. MITRA, D. C. CHOUDHURY,  
(MRS.) T. K. BHAR & A. P. MITRA

*Wireless Laboratory, University College of Science, Calcutta*

Fig. 1 represents the mean hourly values of the penetration frequencies of the E and E<sub>s</sub> regions and the penetration frequencies and virtual heights of region F<sub>2</sub> in graphical form. The figures are obtained from average values of the data taken for each hour of the day for 5 days a week. Fig. 2 gives the prediction of the maximum usable frequencies which can be used for different distances of transmission by reflection at the F region over Calcutta for the month of December 1949. Table I gives the different occasions during routine observation when sporadic E ionization was observed and the values of the corresponding frequencies and heights.

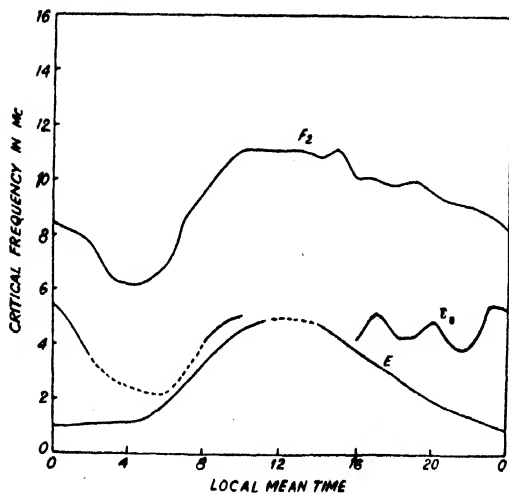
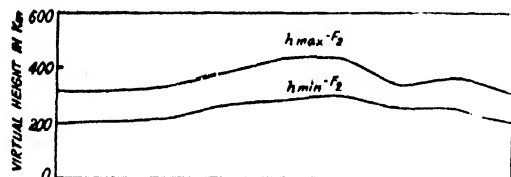
Sporadic E ionization was found to occur less frequently in this month than in the previous one. Absorption of normal E echoes at mid-day was also less. The behaviour of region F<sub>2</sub> was found to be normal in this month.

### TABLE I

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$hE_s$ Km.
September 1949	1	09.00	4.70	120
		17.00	4.50	120
	2	10.00	5.80	135
		18.00	6.25	135
		19.00	6.10	135
		20.00	4.85	120
		21.00	4.40	120
		22.00	4.20	120
		23.00	5.50	120

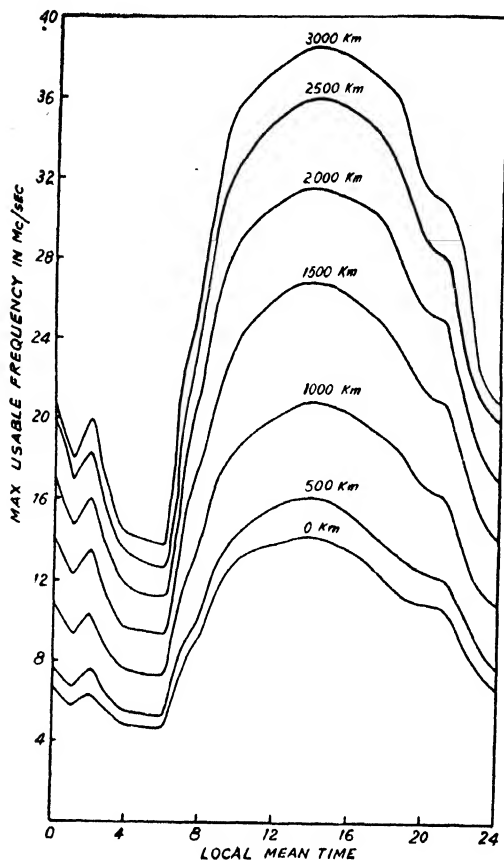
TABLE I—contd.

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$h_{E_s}$ Km.
September 1949	3	00.00	5.35	105
		01.00	4.70	105
		02.00	3.50	90
		05.00	3.10	90
		10.00	4.40	120
		11.00	4.50	120
	8	17.00	6.80	135
		18.00	4.60	135
	9	18.00	3.25	120
		19.00	5.00	120
		20.00	4.00	120
		22.00	3.00	105
	10	08.00	4.00	120
	12	10.00	4.20	120
		11.00	4.40	120
		17.00	4.25	120
		18.00	3.20	120
		19.00	2.25	120
	13	05.00	1.35	105
		06.00	2.10	105
		10.00	4.65	120
		17.00	6.20	135
		18.00	4.70	135
		19.00	4.20	120
		20.00	6.00	120
		21.00	4.00	120
	14	10.00	6.55	150



(5 HR. 54 MIN. AHEAD OF G.M.T.)

FIG. 1—SEPTEMBER 1949.



AT POINT OF REFLECTION.

(5 HR. 54 MIN. AHEAD OF G.M.T.)

FIG. 2—PREDICTED M.U.F. VIA  $F_2$  LAYER, DECEMBER 1949.

TABLE I—contd.

MONTH & YEAR	DATE	HOUR	$f^{\circ}E_s$ Mc.	$h_{E_s}$ Km.
September 1949	15	11.00	4.40	135
		18.00	3.25	135
		21.00	4.60	135
		22.00	4.65	135
	19	21.00	3.00	120
	20	09.00	5.10	135
		10.00	4.75	135
	21	17.00	4.95	135
		18.00	5.20	135
	23	18.00	4.20	135
		19.00	4.00	135
	24	08.00	4.00	120
		09.00	4.50	135
	26	17.00	5.00	135
		18.00	4.90	135
		19.00	4.00	120
	28	18.00	4.20	120
		17.00	4.00	120

## United Nations Research Laboratories

**T**HE United Nations Economic and Social Council, at its 33rd and 34th meetings in 1946, passed the following resolution :

“ The Economic and Social Council, considering : (1) That a certain number of research activities can only be conducted in a rational manner on an international scale ; (2) that many branches of scientific research connected with the promotion of human knowledge, and especially with public health, would yield considerably more effective results if they were conducted on an international plane, invites the Secretary-General to consult UNESCO and other specialized agencies concerned and to submit to the Economic and Social Council a general report on the problem of establishing United Nations Research Laboratories.”

Following this decision, the Secretary-General opened an enquiry on the problem of establishing “ United Nations Research Laboratories ”. In accordance with the terms of the resolution, it was felt that in addition to UNESCO, which was specifically mentioned in the resolution, the following agencies were to be considered as specially interested in this problem : the World Health Organization, the United Nations Food and Agricultural Organization, the International Labour Organization and the International Civil Aviation Organization. The enquiry was subsequently extended to international and national organizations and eminent scientists.

Though opinions differ as to the specific purpose to be served by the U.N. laboratories, the majority of those consulted were in favour of the principle of international research and the setting up of a system of U.N. research laboratories. This view is based on the following considerations : (1) Small countries find it impossible, for financial reasons, to carry out research in certain fields like astronomy, thermodynamics and geophysics which require expensive equipment and specialized experts ; (2) the U.N. research laboratories would be able to work much faster, disseminate information more efficiently than national institutions and eliminate wasteful repetitions ; (3) certain problems of international interest could not

be investigated within the confines of any given state ; Arctic and oceanic studies fall in this class ; and (4) research in fields that are of immediate concern to all nations, or to the majority of nations, such as agricultural science, medicine and public health are facilitated by the establishment of U.N. research laboratories.

The arguments against the establishment of international laboratories include the following : (1) The expense involved would be considerable and the establishment of central laboratories would stifle the development of existing research institutions ; in this connection the Economic and Social Council adopted the view put forward by India that nothing should be done that might hinder, in any way, the development of national laboratories ; (2) it would be difficult to secure the necessary talent for the international research centres ; this view is in sharp contrast with that expressed by small nations who feel that international laboratories under the United Nations would provide the much-needed training facilities and thus help them expand their corps of experts ; (3) there is not enough scientific co-operation in the world at present to justify the hopes that nations might allow the U.N. laboratories to work freely ; this weakness of current scientific effort appeared more as an argument for rather than against further efforts ; (4) certain researches were the product of individual reflection ( mathematical researches, for instance ) which could possibly be injured by restrictions imposed by collective action ; even here particular aspects of the subject required the use of extremely complicated and costly computation apparatus which is not within the means of all countries and so justifies the establishment of international centres.

The general consensus of opinion being overwhelmingly favourable to the principle of international research, the formation of a small committee of experts to examine, in consultation with the specialized agencies of the U.N., the question of establishing international research laboratories, and the advisability of, and appropriate procedure for, convening an international conference was recommended in a resolution adopted by the Council on 9th August 1948. This resolution



was communicated by the Secretary-General in September 1948 to the member states and the specialized agencies of the U.N. and to other international organizations expressing the Council's desire to be apprised of their findings on the problem of establishing U.N. research laboratories. By July 1949, 24 governments had sent in their replies.

In India, a sub-committee constituted by the Advisory Committee for Co-ordinating Scientific Work examined the question as to which of the U.N. research laboratories and observatories could be located in India. The following laboratories and research stations, in the order of priority, were recommended :

*Laboratories* — (1) A laboratory of oceanography and fisheries ; all the conditions required for development of these subjects, namely availability of a good harbour (at Port Blair), opportunities for collaboration with the Nutritional Laboratory and the Food Technological Institute, availability of all types of water, shallow, deep, very deep, etc., and a central position in Asia are all available in India ; (2) nutrition and food technology laboratory to collaborate with the already existing Nutrition Research Laboratory at Coonoor and the Central Institute for Food Technology at Mysore, on the one hand, and the international chain of such laboratories, on the other ; (3) computing laboratories ; there were important centres of work in both mathematical physics and mathematical statistics in India to justify the location of such a laboratory.

*Research Stations* — (1) Institute for research on tuberculosis ; (2) study of life and resources of humid zones ; India can offer the proper background for such an institution and would welcome the setting up of a zonal station ; (3) an institute for human biological and genetical analysis ; the ancient land of India has been the home of many races and can offer unique facilities for studies in this field ; (4) institute and stations for the study of arid zone ( desert and arid tropical zones ).

Several governments transmitted comments from their national scientific establishments. The Athens National Polytechnic Institute felt that there should be a clearing house of information. The Norwegian Research Council was in favour of setting up international laboratories to deal with oceanography and fisheries. The Royal Society

of London considered that the creation of new research institutes in certain regions was necessary for the advancement of such subjects as marine biology of the Pacific and Indian Oceans. The Norwegian institutes recommended the establishment of a meteorological institute. The National Research Council of the U.S.A. recommended three projects : (1) an international computation centre ; (2) an institute of geo-medicine ; and (3) a research institute for social sciences. The International Astronomical Union also preferred the creation of a U.N. computation laboratory to serve all sciences. The Austrian Government recommended the creation in Austria of an institute of higher biological studies. The opinion that no new institution should be established which does not meet a need clearly expressed by the U.N. as a whole was shared by a number of member states.

The UNESCO report on the subject enunciated certain general principles which should govern the establishment of international laboratories and observatories. The following were recommended as first priority projects : (1) an institute for the study of chemistry and biology of the self-reproducing substances, including cancer research ; (2) a chain of laboratories in nutritional science and food technology ; (3) study of life and resources of the humid equatorial zone ; and (4) one or more institutes of oceanography and fisheries in Asia.

Recently, the United Nations and the UNESCO have named a Committee of Experts to advise them regarding the steps to be taken to implement the various recommendations. The Committee, which has just concluded a meeting at UNESCO House in Paris, has recommended that top priority be given to : (1) a central institute of computation and statistical consultation ; (2) an institute of neurophysiology ; and (3) an institute of social studies. Other fields of science recommended as second priorities are : (1) a meteorology institute to serve the needs of aviation, agriculture, weather forecasting and radio industry ; (2) an astronomical laboratory to analyse photographs made by observatories throughout the world ; (3) an arid zone institute to study the most effective methods of turning the world's deserts to productive uses and the utilization of solar energy ; and (4) a biochemistry laboratory to study self-reproducing substances such as viruses and genes.

# Scaler Units for Counting Pulses from Geiger-Mueller Counters or Ionization Chambers

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## 1. Introduction

**N**UCLEAR or radioactive phenomena induce electrical impulses in Geiger-Mueller tubes and ionization chambers and they are rendered easily detectable by means of uni- or multi-stage RC coupling electronic circuits. These impulses, being originated by radioactive phenomena, are of random distribution and consequently there are large fluctuations in the intervals between them. The shortest time interval being the most probable, many of the impulses come too close together to be resolved by the electrically operated mechanical recorder so that even with a fast recorder, capable of counting 5,000 periodic pulses per minute, there is an appreciable counting loss even at 50 randoms<sup>1</sup>. At a high impulse frequency, the counting loss will be considerably great. In any case, it is essential, therefore, to insert a unit or a system of units in order to scale down the rate of impulse frequency to a rate that is capable of being recorded by the electrically operated mechanical counter without appreciable error. These units or systems of units used to scale down the rate of impulse frequency by a fixed ratio are known as scaling circuits or simply scalars. The scalars are important items of the equipment of experimental research in nuclear physics and radioactivity.

## 2. Classification of Scalers

There are two principal groups of scalars, the one operating on the power-of-two principle and the other, which is of recent origin, operating on the principle of decimal system of scaling. Of the first group there have been two types of scaling circuits, the one using mercury thyatron tubes based on the well-known design of the thyatron scale-of-two of Wynn-Williams and others<sup>2</sup> or a circuit employing 885 Argon-filled tubes based on the design of Shepherd and Haxby<sup>3</sup> and the other a form of multi-vibrator circuit<sup>4</sup> of hard vacuum tubes adopted for use in scaling circuits. The other group, which

operates on the principle of decimal system of scaling, is of two different types, one employing a ring-of-ten circuit or a combination of a ring-of-five circuit with that of a scale of two<sup>5</sup>, and the other type, employing three standard scale-of-two units and one modified scale-of-two unit, serving as a trigger circuit, the coupling between the first scale-of-two unit and the second unit being via the modified scale-of-two unit<sup>6</sup>.

## 3. Thyatron Scale-of-two

A scale-of-two consisting of symmetrical circuit employing two thyratrons was designed and constructed by Wynn-Williams<sup>2</sup>. In this arrangement, shown in Fig. 1, when one thyatron flashes, its grid loses control and positive pulse applied at the input causes the other thyatron to flash. Hence its anode voltage drops from supply voltage (200 volts) to break-down (flashing) voltage (15 volts). This drop of potential is transmitted to the anode of the first thyatron which drops from 15 volts to approximately -170 volts, extinguishes the flash and the anode potential then rises to the supply voltage. In this way the impulse renders a transfer of a flash from one thyatron to the other. The rise of anode potential of one of the thyratrons, which takes place after the flash is extinguished, is used as an impulse to operate the next unit.

This type of scaler is used with advantage for many purposes, especially in cosmic ray

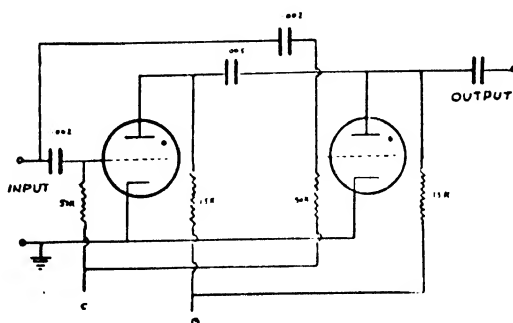


FIG. 1 — THYATRON SCALE-OF-TWO.

studies. Its speed, however, being limited by de-ionization time of thyratrons is not satisfactory for high counting rate. The resolution time of these circuits being long, if the impulses arrive at comparatively short intervals, which occur in the case of impulses of random distribution even of low counting rate, the circuit may be rendered inoperative. These limitations, inherent in the mercury thyatron tube scaling system, which forms a serious handicap to high rate impulse frequency measurements, are overcome to some extent in the counter circuit employing type 885 argon-filled tubes, because the operation of these gas-filled tubes, unlike that of mercury thyratrons, is independent of the temperature and also their de-ionization time is shorter than that of mercury thyratrons. This circuit, however, has one disadvantage — it involves the use of coupling tubes for arresting the large negative pulse and thus preventing the occurrence of doubling.

#### 4. Argon-filled Tube Scalers

A scaling circuit, based on the power-of-two principle, employing 885 type of argon tubes was designed and constructed by Giarratana<sup>7</sup> on the principle of the power-of-two. Its operative process is similar to that of the thyatron scale-of-two. The circuit is stable and reliable and is obviously much simpler than the Shepherd Haxby circuit inasmuch as coupling tubes have been eliminated. With this arrangement of scale-of-eight, it is possible to obtain records up to speeds of nearly 2,000 random pulses per minute without much appreciable loss. The time constant of the input pulse, however, cannot be made adequately small so as to choose the resolving time of the first and the subsequent stages sufficiently small because of the coupling condensers of high capacitance. Therefore, either when the interval between impulses is too small, which does happen in the case of random ones, or when their counting rate is too high, these impulses may easily be missed, resulting in appreciable counting loss.

#### 5. Advantages & the Principle of Operation of Vacuum Tube Scaling Circuit

The difficulties associated with mercury-thyatron scaling circuits and argon-filled tube scaling circuits are overcome in the scaling circuits of hard vacuum tubes inasmuch as they have high speed and stability which render comparatively short resolving

time possible. Vacuum-tube scaling circuits, broadly speaking, are based on special application of the principle of retro-action to two vacuum tubes, so as to produce two states of stable equilibrium. In one state, one tube of the pair has low impedance and, therefore, is conducting, while the other has high impedance and consequently is blocked. In the other state, on the other hand, the situation is reversed so that the former one is non-conducting and the latter is conducting. In a design involving such a principle of operation, scaling down the rate of random impulses to a half is accomplished inasmuch as at each incident pulse the circuit is tripped from one stable state to the other.

Existing scaling circuits employing vacuum tubes are all modifications of the outlined scheme with slight differences in the constructional and certain operational details. The constructional details influence, to a considerable extent, the stability, reliability and the efficiency of a scaling circuit. There have been two kinds of circuits employing vacuum tubes, one with triode valves and the other with pentode valves.

#### 6. Triode Vacuum Tube Scaling Circuit of Lewis

Lewis<sup>8</sup> has designed a scaling system based on a symmetrical circuit of two vacuum triode valves having two states of stable equilibrium. When a positive impulse is applied to the line between the bias rectifiers connecting the grids of the two valves, the circuit is triggered from one stable state to the other. A continuance of the relative change of the grid potentials is maintained by the inductance of the choke included in the circuit so that the stable state which results is not the one from which the unit started. Thus, successive impulses drive the unit alternatively from one state of stable equilibrium to the other. A scale-of-eight comprises three of these scale-of-two units, followed by a thyatron-operated mechanical recorder.

A scale-of-eight, employing hard vacuum valves, is expected to achieve a much higher counting rate than the one employing mercury thyratrons. But Lewis's scale-of-eight has a maximum counting rate which is not greater than that obtained by the thyatron scale-of-eight. The advantage of high speeds obtainable by hard vacuum valves could not be turned into good account because of the

restrictions associated with the coupling condenser and the choke. The circuit is stable and satisfactory for many purposes. It does not involve complex coupling units besides simple condensers for its operation. The employment of specially constructed grid bias rectifying units and chokes, however, makes the construction of the circuit far from simple.

## 7. Triode Vacuum Tube Scaling Circuit of Lifschutz & Lawson

A scaling circuit employing triode valves, in which the high speed of such vacuum tubes is made use of for obtaining high counting rates, has been designed and constructed by Lifschutz and Lawson<sup>9</sup>. A circuit (FIG. 2) employing two triode vacuum valves forms a scale-of-two unit in which pulses are applied to the valve grids in parallel through resistance-capacity coupling. The first valve of the pair is direct-coupled to the other by a resistor from its plate to the grid of its mate and similarly the second one is direct-coupled to the first. Hence the system is symmetrical. The symmetrical direct coupling causes the circuit to have two states of stable equilibrium. Input pulses trip the circuit from one state of stable equilibrium to the other, causing an increase or decrease of the plate potential of each valve at alternate pulses. An electrical division by two is accomplished by the transmission of the plate potential changes of the second valve to a thyratron-operated mechanical recorder which operates only at the positive changes.

In a multiple-stage scaling circuit, the output pulses of the second valve of each stage are fed by RC coupling to an additional triode valve which is biased past cut-off so that only every other pulse is transmitted to the next scale-of-two. A scale-of-sixteen built by Lifschutz and Lawson comprises four such triode scale-of-two systems with triode coupling elements followed by an output stage operating a mechanical recorder. This circuit has a high rate of impulse frequency counting and seems to be stable and reliable. It cannot, however, be deemed to be simple owing to the complex triode valve coupling units. In a scaling circuit of this type, if simple triode vacuum tubes are employed, the oscillations of the one may be picked up by the rest of the stages. It is also sensitive to external disturbances. These defects, which cause spurious countings and render the counting records unreliable, can be eliminated by covering the components

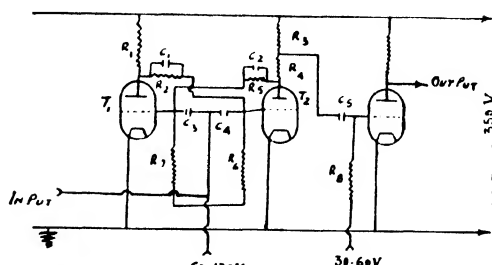


FIG. 2 — TRIODE VACUUM TUBE SCALE-OF-TWO CIRCUIT.

$C_1 = C_2 = C_3 = C_4 = C_5 = 25 \mu\text{F}$ ;  $R_1 = R_8 = 0.05 \text{ M}\Omega$ ;  $R_2 = R_6 = 0.04 \text{ M}\Omega$ ;  $R_3 = 0.03 \text{ M}\Omega$ ;  $R_4 = 0.02 \text{ M}\Omega$ ;  $R_5 = R_7 = 0.1 \text{ M}\Omega$ .

of each stage by a separate grounded metal shielding or by employing triodes of the metal shell self-shielding type.

## 8. Double Triode Vacuum Tube Scale-of-two

The circuit shown in Fig. 3 has been developed at the Department of Physics, University of Michigan. It is based on the principle of power of scale-of-two, employing single double triode vacuum tube instead of two single triode vacuum tubes as in the circuit of Lifschutz and Lawson. One triode of the double triode is direct-coupled to the other triode by a resistor from its plate to the grid of the other one, and likewise the second one of the double triode valve is direct-coupled to the first triode of the double triode vacuum tube so as to form a symmetrical system having two states of stable equilibrium. The design of this circuit is similar to that of Lifschutz and Lawson except for certain constructional details. The modification in the constructional details of this circuit is initiated by the fact that in this case, a single double triode vacuum tube is employed instead of two triode valves. Owing to the use of improved type of double triode vacuum tube and other components, this circuit allows higher resolution and better reliability than that of Lifschutz and Lawson.

In a multiple power of scale-of-two, the output of the second half of each stage is transmitted by RC coupling to the grid of an additional triode vacuum tube which is biased past cut-off so as to permit transmission of every other pulse to the next stage.

A scale-of-sixty-four comprising six units of this type of scale-of-two, joined together in cascade and each unit provided with a neon lamp across the right-hand anode of the

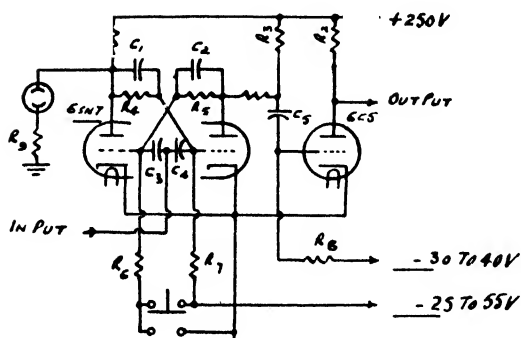


FIG. 3 —  $C_1=C_2=100 \mu\text{F}$ ;  $C_3=C_4=C_5=50 \mu\text{F}$ ;  $R_1=R_2=R_3=R_4=R_5=0.05 \text{ M}\Omega$ ;  $R_6=0.03 \text{ M}\Omega$ ;  $R_7=R_8=0.1 \text{ M}\Omega$ ;  $R_9=1 \text{ M}\Omega$ .

double triodes in each of the six units for interpolation, was employed by the present author<sup>10</sup> for the measurement of beta-disintegration energies of antimony<sup>124</sup>, tantalum<sup>182</sup>, tungsten<sup>185</sup> and iridium<sup>192</sup>. The first unit of scale-of-two has a resolution time of less than 2 microseconds. The scale-of-sixty-four circuit with a mechanical recorder having a speed of 20 counts per second has an efficiency of 99 per cent at a counting rate of 1000 counts per minute.

#### 9. Alfven's Triode Vacuum

##### Tube Scale-of-two

The circuit of scale-of-two employing triode vacuum tubes is shown in Fig. 4. It was designed and constructed by Alfven<sup>11</sup>. It is simpler than the other designs described before. The principle of its operation is similar to that of the other circuits of scale-of-two employing triodes. It, however, differs from them in certain constructional details. Unlike Lewis circuit, neither cuprous oxide rectifiers nor chokes are employed in this circuit. An addition of two or more of these scale-of-two units of this design into a single system of multiple stages does not seem to involve triode valve coupling systems between the stages, unlike the multiple-stage scaling circuit of Lifschutz and Lawson.

This circuit, like the former, has two states of stable equilibrium. In one state, one tube is blocked and the other is conducting, while in the other state the situation is reversed. Positive pulses, applied to the line between the "condenser-resistance grid-biases coupling", trip the circuit from one state of stable equilibrium to the other and the resulting positive pulse from the plate through an auxiliary by-pass CR coupling drives the circuit in an opposite state after

the tripping pulse is over. It is these conditions that render an electrical division by two possible. Impulses are transmitted from the plate of one of the valves through a RC coupling to a thyatron circuit which operates a mechanical recorder. A further scaling may be obtained by adding two or more scale-of-two units by directly connecting the output of one stage to the input of the next without coupling units.

Alfven has mentioned neither the highest obtainable counting rate nor the efficiency of its performance in his publication. With his counter circuit, he obtained records of about 200 counts per minute and this rate obviously is too low for nuclear measurements. He points out, however, that higher counting rate may be obtained by this unit.

#### 10. New Model Double

##### Triode Valve Scale-of-two

One of the latest models of scaler, employing a single double triode and one double diode, was designed and constructed by Higinbothams, Gallagher and Sands<sup>12</sup>. The circuit, shown in Fig. 5, is based on Eccles-Jordan circuit<sup>13</sup>, modified and symmetrically coupled to a single source of triggering by means of diodes. The circuit is triggered alternatively from one state of stable equilibrium to the other by successive identical triggers. The two diodes used in the circuit ensure a definite switching over from one state of stable equilibrium to the other and prevent any triggering operation by pulses of wrong polarity. The two halves are mutually direct-coupled by resistors from the plate of the one to the grid of the other, and similarly from the plate of the second to the grid of the first as in the double triode vacuum tube scale-of-two circuit. The symmetrical coupling induces a large measure of stability which is enhanced by the self-

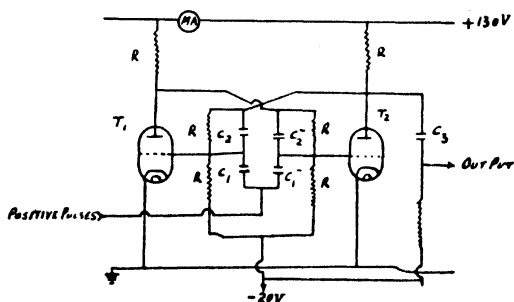


FIG. 4 — ALFVEN SCALE-OF-TWO.  
 $T_1-T_2$ , PHILIPS B 2038;  $R=0.2 \text{ M}\Omega$ ;  $C_1=C_2=C_3=1000 \text{ CM}$ ;  $C_4=C_5=500 \text{ CM}$ .

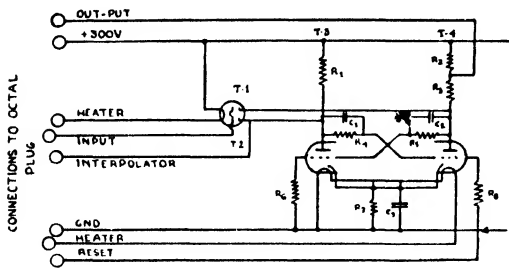


FIG. 5 — T-1 AND T-2-6H6, T-3 AND T-4-6 SNT.

$R_1=0.02 \text{ M}\Omega$ ;  $R_2=0.05 \text{ M}\Omega$ ;  $R_3=0.15 \text{ M}\Omega$ ;  
 $R_4=R_5=0.2 \text{ M}\Omega$ ;  $R_6=R_7=0.1 \text{ M}\Omega$ ;  $R_8=0.01 \text{ M}\Omega$ ;  
 $C_1=C_2=50,4.4 \text{ F}$ ;  $C_3=0.01 \text{ }\mu\text{F}$ .

biasing arrangement in the cathode circuit of the double triode. The symmetry of the arrangement which causes the circuit to be driven by successive impulses from one state of stable equilibrium to the other together with the unilateral switching action by the two diodes accomplishes an electrical division into two.

A scale-of-sixty-four, comprising six units of this model of scale-of-two was built by the authors. The first scale-of-two unit, employing 6 SN7 type, has a resolving time of 2 to 5 microseconds depending upon the method of joining the different units while the subsequent units, employing 6 SL7, have a resolving time of 20 microseconds. The circuit is simple, requires no adjustment and operates reliably.

### 11. Pentode Tube Scalers

Scalers employing pentode tubes were developed by (1) Stevenson and Getting<sup>14</sup>, (2) Reich<sup>15</sup> and Frisch<sup>16</sup>. These scaling circuits, like the triode vacuum tube scaling systems, are designed so as to apply the principle of retro-action to two vacuum tubes. Their constructional and operational details, however, differ to a great extent from those of triode vacuum tube scaling circuits. With pentode vacuum tubes, a thorough control over the operation of the scaling system, so as to obtain high efficiency and reliability, is more easily obtainable than with triode vacuum tubes. With pentode vacuum tube scaling circuits, under certain conditions which are easy to realize, metal screening elements of the tubes and the grounded metal shielding walls between the stages may be omitted without impairing the insensitivity of the circuit to external disturbances.

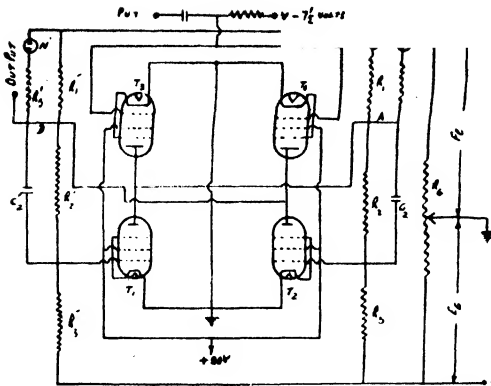
### 12. Pentode Tube Scaling Circuit of Stevenson & Getting

Stevenson and Getting<sup>14</sup> devised and constructed a circuit employing pentode tubes for scaling down counting rates. The circuit shown in Fig. 6, in its essentials, consists of two pentode vacuum tubes with a symmetrical arrangement of two sets of resistors across which a combination positive plate supply and negative grid bias is applied. The circuit has, of course, two states of stable equilibrium; in one state one tube is conducting and the other tube is non-conducting, while in the other state the position is reversed so that the latter is conducting and the former is blocked. Two additional pentode vacuum tubes and condensers for cross coupling are employed in the circuit. The grids of the additional tubes are normally negatively biased and the plates of the pair are connected to those of the scaling pair. A positive pulse, applied to the grids of the additional pair, trip the circuit from one state of stable equilibrium to the other, so that the initially conducting becomes non-conducting and the initially non-conducting becomes conducting. The next incident pulse which enters the second tube of the additional pair, connected to the present conducting tube, trips the principal scaling pair back to the original position. Thus, successive incident pulses repeat the process. The resulting alternate positive potential changes of either one of the tubes of the principal pair are transmitted directly to the circuit of the mechanical recorder.

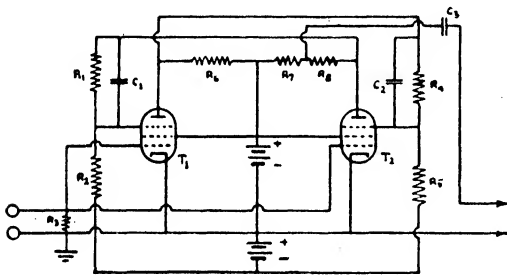
A scale-of-thirty-two, comprising five of scale-of-two units, directly connected in series, finally followed by an output stage, operating a mechanical recorder, was constructed by Stevenson and Getting<sup>14</sup>. The scaling circuit is efficient, stable and reliable and has a high resolving power. It is not simple and employs four pentode vacuum tubes for each stage. The circuit is not insensitive to external disturbances and hence the sensitive parts of the circuit need adequate shielding.

### 13. Reich Pentode Vacuum Tube Scaling Circuit

A scale-of-two circuit which is a modification of a trigger circuit<sup>17</sup> whose operation is based upon the principle of retro-action between two tubes, has been described by Reich<sup>15</sup>. The circuit, shown in Fig. 7, is simple in design and, unlike the circuit of Stevenson and Getting, requires only two pentode vacuum tubes.

FIG. 6 —  $T_1, T_2, T_3$  AND  $T_4 = \text{TYPE 57}$ .

$R_1 = R_2 = R_3 = R_4 = 100,000 \Omega$ ;  $R_5 = R_6 = 300,000 \Omega$ ;  $R_7 = R_8 = 500,000 \Omega$ ;  $C_1 = C_2 = 0.00025 \text{ HF}$ ;  $C_3 = 0.000025 \text{ HF}$ ;  $N$  AND  $N^1 = 10 \text{ KATT NEON LAMPS}$ .

FIG. 7 —  $T_1 = \text{TYPE 57}$ ,  $T_2 = 6J7$ .

$R_1 = R_2 = R_3 = R_4 = R_5 = 0.25 \text{ M}\Omega$ ;  $R_6 = 0.055 \text{ M}\Omega$ ;  $R_7 = 0.005 \text{ M}\Omega$ ;  $R_8 = 0.05 \text{ M}\Omega$ ;  $C_1 = C_2 = C_3 = 50 \mu\text{F}$ .

An operation of scaling is based upon triggering action. In this circuit the coupling is obtained by means of suppressor grids through a symmetrical arrangement of condenser-resistance by-passes. Input negative voltage pulses are applied to the control grids. The screen grids are normally at a positive potential, and the suppressors at cathode potential. The circuit is very sensitive to negative voltage pulse, and is altogether insensitive to pulses of positive potential. A current transference from the tube 1 to the tube 2 presents a negative pulse, while its transference from tube 2 to tube 1 a positive pulse. It is because of these characteristics that, firstly, an electrical division into two may be accomplished and, secondly, one stage may be coupled to the next without the use of coupling units and rectifiers.

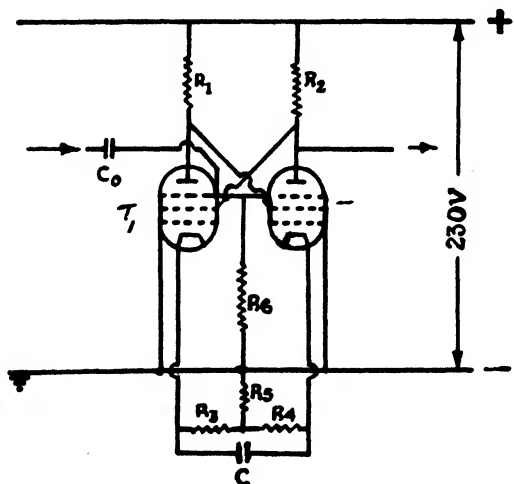
The design of this scaling circuit is simple. It operates on a total voltage of about 45

volts. In this circuit the input pulses are applied to control grids which are by far the more sensitive than the rest. Hence, it has a high degree of sensitivity which may be turned to useful account in cases where the amplitude of the incident pulses is very small. In cases where the electrical counter pulses are amplified, equalized with amplitude rightly adopted, such a high degree of sensitivity is not of any special value. This circuit is not insensitive to external disturbances. Therefore, it may easily pick up stray electrical oscillations and thus may give spurious counts. To eliminate these defects, the tubes and the sensitive components are to be adequately screened by grounded metal shields and the components of each stage are to be shielded from those of every other stage by grounded screening metal walls.

#### 14. Frisch Pentode Vacuum Tube

##### Scale-of-two

A pentode vacuum tube scaling circuit can be rendered practically insensitive to external radio frequencies and other external disturbances by keeping the control grids at earth potential. In this case the triggering pulses which trip the circuit from one stable condition to the other are applied to the suppressors. A scale-of-two with these modifications and with certain changes in the constructional details was developed by Frisch<sup>16</sup>. The principle of this scale-of-two circuit is not different from that of other vacuum tube scaling circuits. It differs from

FIG. 8 —  $R_1 = R_2 = 0.1 \text{ M}\Omega$ ;  $R_3 = R_4 = 600 \Omega$ ;  $R_5 = R_6 = 1 \text{ M}\Omega$ ;  $C_0 = 0.0001 \mu\text{F}$ ;  $C = 2 \mu\text{F}$ .

the rest in the constructional details which influence the operation and the efficiency of the circuit. The circuit, shown in Fig. 8, consists of two pentode vacuum tubes, Sylvania 57 type, the coupling between them, essential for retro-action, being through screening grids and the pulses being transmitted to the suppressors in parallel.

The current through the resistor  $R_1$  is the plate current of the tube I and the screen current of the other, while that through the resistor  $R_2$  is the plate current of the tube II and the screen current of its mate. With a condenser  $C$  in parallel, the resistors  $R_3$  and  $R_4$ , separately connected to the cathode of either one of the valves, the two scaling tubes are individually self-biased, while with the resistor  $R_5$ , the pair is self-biased in common. The symmetrical arrangement of the direct-coupling permits the circuit to have two states of stable equilibrium; in one state one tube is conducting and the other is non-conducting while in the other the condition is reversed.

A negative pulse applied to the suppressor trips the circuit from one state of equilibrium to the other. Each time the circuit is triggered by an input pulse, the current released through the individual self-biasing system with a condenser in parallel, provides for a time,  $t = (R_3 + R_4) \cdot C$ , a bias to the control grid in excess to the common bias furnished by the current through the resistor  $R_5$ . The time constant of the individual self-biasing units with the condenser in parallel  $(R_3 + R_4) \cdot C$  is so chosen as to let the additional bias survive the triggering pulse. The extra negative bias on the control grid, which lasts longer than the triggering pulse, drives the circuit into the reverse state after the pulse is over. The transmission of successive pulses of negative potential repeats the process, producing in each tube plate alternate positive and negative potential changes. A transmission of these potential changes of either one of the tubes into a thyratron-operated mechanical recorder which is sensitive only to the positive potential pulses, accomplishes a scaling down of the counting rate to half the value.

The circuit is quite sensitive to negative pulses. Pulses of positive potential are, however, ineffective as they are absorbed by electron current. It is because of these characteristics that two or more of these systems may be joined in series without coupling vacuum tube units to obtain multiple power-of-two circuit. Frisch described

a scale-of-eight circuit comprising three such scale-of-two units, joined together in series and followed by a thyratron-operated mechanical recorder. In this system the alternate negative plate potential changes of one of the tubes of the preceding unit are directly transmitted to the one immediately succeeding it.

Frisch scale-of-two is simple in design and easy to construct. The circuit can be brought into working order within a reasonably short period. It does not need coupling valves or other complicated coupling units. It has, above all, the advantage over others of being practically insensitive to external disturbances and yet adequately sensitive to tripping pulses of comparatively low potential.

### 15. Pentode Vacuum Tube Scale-of-sixteen

A circuit of pentode vacuum tube scale-of-sixteen, involving such modifications in the constructional details which render it quite sensitive to electrical counter impulses but practically insensitive to external disturbances, was constructed by the author<sup>18</sup> for using it in the measurement of the intensity distribution with momentum of beta rays of uranium  $X_1^{19}$  and radioactive isotope of gold<sup>20</sup>,  $^{198}\text{Au}$ . The circuit, shown in Fig. 9, which is based upon the Frisch scale-of-two, comprises four of these scale-of-two units, placed side by side connecting + to +, - to -, and output to input, followed by a thyratron-operated mechanical recorder with an extra pentode valve operating visual indicator for interpolation.

The operation of the scaling circuit is determined by the amplitude, the resolving time and the abundance of higher harmonics of the input pulses. Pulses of very low amplitude are incapable of tripping the circuit from one state of equilibrium to the other. The amplitude should be adequately large, and for the range of such amplitudes of input pulses, the circuit must be provided with a balancing self-biasing system common to the pair of scaling tubes. Pulses of much larger amplitude which exceed the limit, knock down the individual self-bias in excess to the one common to the scaling tubes with the result that the system is not forced to the reverse state of equilibrium after the pulse is over. Therefore, the circuit, though operates, is incapable of proper scaling action.

Since the individual self-bias, in excess to the one common to both, should survive by tripping pulse in order to urge the system to the reverse state of equilibrium after the





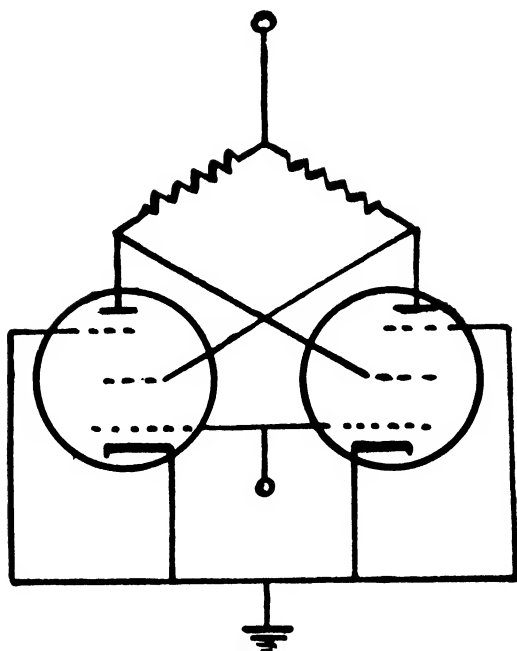


FIG. 10

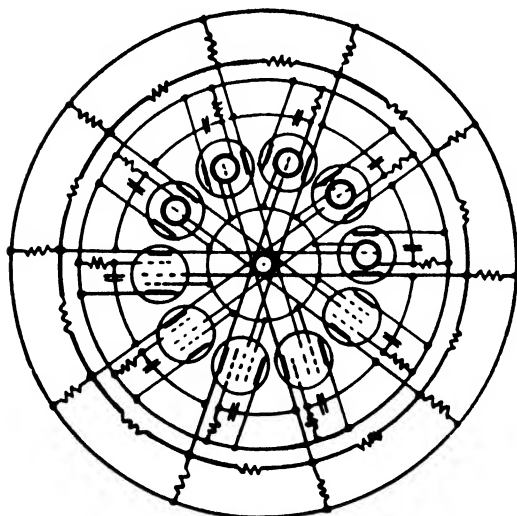


FIG. 11

of limited amplitude which put the circuit successively through its ten states of stable equilibrium producing negative pulses at the plates of the off-tubes only. When a positive pulse is applied to the common control grids, four of the off-tubes and one of the on-tubes receive a negative pulse on their suppressor grids. The off-tubes remain unaffected, but the first of the series of on-tubes is turned off while its partner on the

opposite side of the circle in the series of off-tubes is turned on because of the screen plate connections. Thus, each successive input positive pulse advances the pattern in ten successive steps to complete each round of the ring-of-ten circuit, resulting in scale down action on power-of-ten principle.

The ring-of-ten circuit admirably eliminates additional arithmetical manipulation associated with the power-of-two circuits in the counting rate measurements. They, however, cannot be made very fast because of the limitations inherent in the essential components. Another serious disadvantage which these circuits have is that the components have to be critically adjusted for bringing it to operation.

#### 17. Double Triode Vacuum Tube Scale-of-ten Circuit

The difficulties associated with the ring-of-ten circuits are overcome in a new design of scale-of-ten circuit developed by Taylor<sup>21</sup> and also independently by Rotblat and his collaborators<sup>6</sup>. These two scalars operate on similar principles. It is, however, considered that the circuit designed by the latter author permits a greater resolving power and better reliability than that suggested by the former one since the employment of diodes as coupling units ensures unilateral operation and allows a greater tolerance of components.

The device described by Rotblat and his collaborators<sup>6</sup> consists of four units of which the first three are the scale-of-two units connected in cascade to give a scale-of-eight and the fourth a modified scale-of-two unit which serves as trigger circuit, the coupling being between the first and the second units via the fourth as shown in Fig. 12. In these units, double triodes,  $T_1, T_2, T_3, T_4$ , connected as multi-vibrators, are employed with double diodes,  $D_1, D_2, D_3, D_4, D_5$ , as coupling units. These diodes accomplish unilateral switching action so as to permit triggering action by pulses only of the right polarity and prevent this action by pulses of the other polarity.

These units receive input pulses of negative polarity. When reset relay is put into operation, the right halves of the double triodes are rendered conducting, and this position is designated as the initial state of stable equilibrium. Commencing the run with all units in this initial state, the first pulse will trigger  $T_1$  while the second restores it to the initial state, producing a negative pulse in the output of the first unit which will be an input pulse of the second unit via

the diodes  $D_5$  and  $D_2$ . The fourth unit, of which  $D_5$  is the coupling element, will be unaffected by this output pulse inasmuch as this unit, which is in a state of equilibrium, can be triggered by the pulses coming from third unit via  $D_4$ . The second unit will operate in the same way by the third and fourth input pulses. At the end of the fourth pulse, the third unit will be triggered into non-initial state of stable equilibrium and will continue remaining in the same state with the fifth, sixth, seventh and eighth pulses at the end of which the unit retro-acts to the initial state of equilibrium. In this retro-action, it transmits a negative pulse via  $D_4$  to the fourth unit, thereby tripping it to the non-initial state. The ninth pulse acts on the first unit in the same way as the first. But the tenth operates only on the fourth unit since the upper half of  $D_5$  is cut-off resulting thereby that no pulse is transmitted to the second unit. The tenth pulse will give rise to a negative pulse on the output of the scaler and all units at the same time will be driven to the initial state of equilibrium ready for the next cycle of ten pulses. In this way scaling action in the first power-of-ten is obtained. Higher powers of scale-of-ten circuits comprise of two or more units of scale-of-ten joined together in series.

### 18. Interpolation

The number of counts for a run may not be an exact multiple of the scaling ratio. The actual number of counts scored by the counter over and above the counts recorded by the mechanical recorder is obtained by any one of the methods of interpolation. There are four of these, known as the meter method, the "electron eye" method, the neon lamp method, and the neon indicator method. These methods are useful not only for determining the number of counts scored by the counter over and above the counts indicated by the mechanical recorder but also for observing and checking the individual performance of each stage of the multiple stage scaling system.

*The Meter Interpolation Method* — The meter method transmits a portion of the plate current from one of the tubes in each stage to a common milliammeter. The contribution of the first stage is adjusted by the choice of a suitable resistor to one unit, that of the second to two units, the third to four units and so on, according to the number of scale-of-two stages employed in the multiple stage system. At the commencement of a run, all the tubes feeding the meter are set at a non-conducting equilibrium. Hence the meter reading is zero. After the arrival of the first pulse, the tube of the first stage feeding

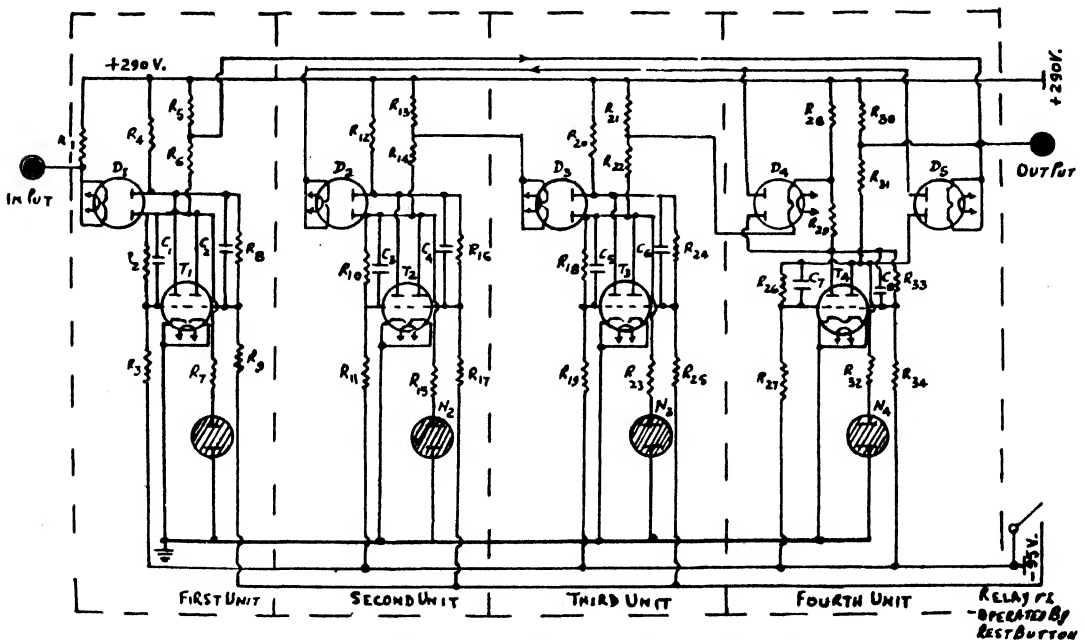


FIG. 12 — CIRCUIT DIAGRAM OF SCALE-OF-TEN UNIT.

the milliammeter becomes conducting so that the meter records one unit. Then, with the second input pulse, while the tube of the first stage connected to the meter ceases to be conducting, the tube of the second stage connected to the meter becomes conducting. The meter, therefore, having drawn from the tube of the second stage twice as much as that drawn from that of the first stage, records two units indicating that two pulses have been scored. Similarly, with successive pulses the meter reading goes up in uniform steps and falls back to zero when the number scored has reached the scaling ratio.

*The "Electron Eye" Interpolation Method* — The "electron eye" method of interpolation employs electron ray tubes, one tube being direct-coupled to each scale-of-two stage. In this method, the number assigned to that scale-of-two stage is added to the reading of the mechanical recorder when the "eye" of the tube belonging to that stage is opened. The additional number of counts scored over the counts recorded by the mechanical counter can be more easily read by the meter method than by the "electron eye" method. This method, however, has the advantage of being extremely fast and of not reacting on the scaling circuit.

*The Neon Lamp Interpolation Method* — This method employs miniature neon lamps in each unit of scale-of-two of the multiple power of scale-of-two unit for interpolation. These lamps are connected across the left-side anodes or right-side anodes of each of the double triodes in each of the multiple units, depending upon the type of circuit connections (see FIG. 3 and FIG. 12). These lamps are lit every time its coupled triode is driven to non-conducting state. These neon lamps correspond to the number of counts 1, 2, 4, 8, 16 and so on, and the sum of these numbers assigned to the neon lamps lit gives the number of pulses scored over and above the number recorded by the mechanical counter. The actual number of counts for a given run is, therefore, the sum of impulse counts scored by the neon lamps lit and the counts recorded by the mechanical counter. This method is very fast and reliable. It does not react on the scaling circuit. In this method, the circuit connections are simple and straightforward.

*The Visual Indicator Method of Interpolation* — The visual indicator method consists of a neon visual tuning tube ("Tuneon", General Electric Company, England), whose

long-wire electrode is placed in the plate circuit of an additional pentode vacuum tube "Sylvania" 57 type, its grid bias being connected through suitable resistors to the plate of one of the tubes of each one of the four scale-of-two stages as shown in Fig. 9. It is essential that these coupling plate resistors from the first to the fourth scale-of-two stage are in the ratio of 8:4:2:1 in order to obtain a glow discharge of the wire electrode growing up in equally measured steps with each input pulse.

At the beginning of a run of the scaling circuit, when all the tubes feeding the grid bias of the additional valve coupled to the visual indicator are set at non-conducting equilibrium state — since there is no excess of bias added to the normal grid bias of the additional valve — the grid potential is a minimum and consequently the plate current drawn by the long-wire electrode is also a minimum. In this stage, therefore, the glow discharge of the electrode, like a spot, is localized at the foot of the wire electrode. As the first input pulse trips the circuit, the valve of the first stage, connecting the grid bias of the additional valve, becomes conducting so that its plate current which passes through the resistor will be an additional bias in excess to the normal bias. Thus successive pulses render the tubes of the successive stages, connected to the grid bias of the valve coupled to the visual indicator, one after another, conducting. Therefore, with each input pulse, the grid bias of the valve, coupled to the visual indicator, is increased in equal steps in excess of the normal bias, and consequently also the grid potential in equal steps, so that with the input of each pulse the length of glow discharge of the wire electrode increases in equal measures. In this way the length of the glow discharge of the wire electrode keeps on increasing in equal measures until the number of counts scored reaches the scaling ratio, when the thyratron flashes and operates the mechanical recorder. Then the tubes of the four scale-of-two stages connected to the grid bias of the additional valve of the visual indicator are once more in the non-conducting equilibrium state, the grid bias and the grid potential run down to their initial values and consequently the glow discharge of the wire electrode falls down to the zero position. If the fall length of the glow discharge of the wire electrode is calibrated into divisions, each division corresponding to the length of the glow due to each

input pulse, the number of counts scored over and above the counts indicated by the mechanical recorder can be directly read from the visual indicator tube divisions.

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# Market Quality of Rice

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**T**ASTE and flavour on which quality of foodstuffs greatly depends are difficult to define and are to some extent based on common beliefs and prejudices. It is generally believed that short duration varieties of rice are poorer in quality than the long duration varieties, because they are assumed to remove less of plant food from the soil. In South India the short duration *Kuruwai* or *Kar* varieties are not valued as much as the long duration *Samba* varieties. Similarly, in Bengal it is held that *Aus* rice (*Indrasail*, *Dudahar*, *latisail*, *jingasail*, *nagra*, etc.) are inferior to *Aman* varieties (*Charnock*, *dhairal*, *Kumari*, *Chulmuri*, etc.). Irrigated rice crop is reported to be poorer in quality than the dry one. Parboiled rice has been proved to be of greater nutritive quality.<sup>1-11</sup> Yet it is considered by some as not "good" though "wholesome". Quality varies also with regions. The long, slender and fragrant rice grown in the United Provinces or the Punjab is not appreciated in South India, while a parboiled, hand-pounded

rice, red or reddish yellow in colour, popular in Bengal, Bihar and Orissa, is not considered good in other parts of Northern India.

With such wide variations in personal factors and the fact that nearly 7,000 varieties of rice are cultivated all over the world under varying climatic conditions and cultural operations and processed to suit different markets and trades, it is not surprising to find in the rice market innumerable trade descriptions, varieties and grades. Rices are fine, medium or bold, raw and parboiled, machine milled and hand pounded. In most of these kinds translucency, fragrance, uniformity in size and shape, age or maturity and cooking quality are some of the more prominent quality factors. Added to this are considerations of a more general nature like the proportion of foreign matter, red grains, damaged or defective grains, etc. A natural translucency and fragrance, which persists even after cooking, as for example, in the *Basmaties* of the United Provinces and the Punjab, *Chimur* in the Balaghat district

TABLE I—QUALITY CHARACTERISTICS OF SOME "SPECIAL GRADE" AGMARK RICES

NAME OF RICE	LOCALITY	TREATMENT	FOREIGN MATTER %	BROKEN GRAINS %	FRAGMENTS %	OTHER RICE GRAINS INCLUDING RED RICE %	DAMAGED OR DISCOLOURED GRAINS %	WEEVILLED GRAINS %	CHALKY GRAINS %	WT. OF 1,000 KERNELS gm.	SIZE OF GRAIN	
											Length mm.	Breadth mm.
<i>Kitchisamba</i> G.E.B. 24	Madras Presidency	Raw, milled	Traces	4.8	0.8	2.0	Traces	Free	Slightly chalky	13.0	5.2	2.1
<i>Nellore Samba</i>	Tanjore and Trichinopoly	"	"	7.6	0.6	3.2	0.5	"	1.6	14.8	5.5	2.3
<i>Krishnakakulu</i>	East and West Godavari	"	"	4.4	1.0	2.8	None	"	0.5	13.9	5.3	2.0
<i>Atragada</i>	Kistna and Guntur	"	"	8.2	2.0	1.6	"	"	Chalky	17.6	5.7	2.4
<i>Dehi Bhogum</i>	"	"	"	5.8	2.0	1.2	"	"	Slightly chalky	14.6	5.6	2.1
<i>Kusuma</i>	"	"	"	11.2	2.6	1.0	"	"	Chalky	18.3	5.9	2.3
<i>Molagolkulu</i>	Nellore	"	"	5.3	0.4	2.0	0.5	"	1.8	15.6	5.2	2.3
<i>Red Strumani</i>	Tanjore and South Arcot	Parboiled, milled	"	3.2	nil	1.2	0.2	"	nil	15.2	4.8	2.6
<i>White Strumani</i>	"	Raw, milled	"	10.4	1.0	1.8	1.0	"	2.2	11.8	3.9	2.6
<i>Kasipichody</i>	West Godavari	"	"	4.2	1.8	2.8	Traces	"	Chalky	9.8	5.5	1.6
<i>Basmati sela</i>	Saharanpur	Parboiled, milled	"	0.8	nil	2.6	0.2	"	1.0	14.5	6.8	1.8
<i>Basmati</i>	Dehra Dun	Raw, milled	"	3.2	"	0.8	0.2	"	nil	14.9	6.8	1.9

of the Central Provinces and *Ambemohar* of Bombay; a suitable colour, white being generally preferred, are other desirable factors of quality in rice.

Rices are usually graded on the above considerations. The quality characteristics of some special grade *Agmark* rices analysed by the writer are summarized in Table I.

It will be seen that the grading is more empirical than scientific. A scientific system of rice grading is still a desideratum.

Generally speaking, quality is also governed by the use to which the rice is ultimately put. The utilization of rice is illustrated in Fig. 1. It is evident that rices required for different uses demand different quality grades. To illustrate: rice required for the laundry or textile industry is of a different quality from that used for seed. Even in the case of human consumption, special quality rices are required for the ever so many preparations in which rice is consumed.

For consumption, as plain boiled rice, which is by far the most outstanding use of rice, quality is generally based on the size and shape characteristics of the grain. The size characteristics of the common rices are illustrated in Fig. 2. Of the several grain sizes illustrated, three important groups are prominently distinguished in the marketing of rice, viz. fine, medium and coarse. Other factors being equal, fineness or slenderness is indicated by a fairly high length to breadth ratio and small size and this singly or in combination with other desirable characteristics places rice high in its market quality.

In the above paragraphs an attempt has been made to define and illustrate the market quality of rice—rice which is at present the most important article of diet in South-east Asia. Rice forms the staple food of nearly half the people of the world, and scientists have not yet devoted to it the attention it deserves. It is hoped a more scientific system of rice grading will be evolved when the exigencies of famine conditions are over and normal marketing of rice is resumed.

#### Acknowledgement

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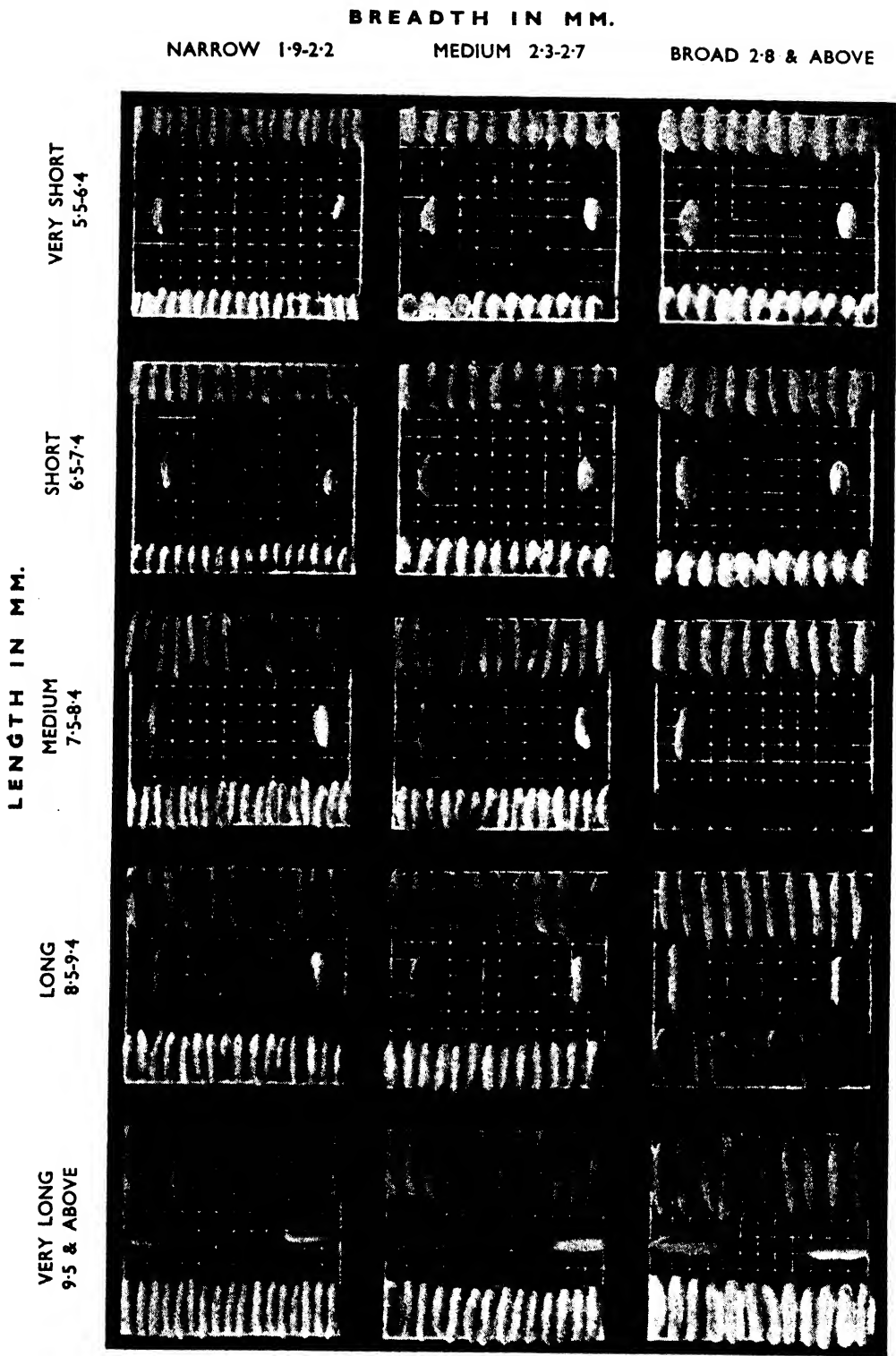


FIG. — 2 GRAIN-SIZE CHARACTERISTICS OF PADDY AND RICE.



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## Development of Powdered-coal Gas Turbine for Locomotive Purposes — a Review \*

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**T**HAT generation of power through steam engines is thermodynamically an inefficient process has been long recognized.

This fact is well brought out in the remarks of Redtenbacher, made more than one hundred years ago, namely "the fundamental principle of the generation and the use of steam is wrong. It is hoped that steam engines will disappear in a not far distant future, as soon as we know more about the nature and the effects of heat."

Even under the best conditions of feed water heating, regeneration, etc., central steam plants utilizing superheated high-pressure steam have attained only 30 per cent thermal efficiency. This efficiency falls to about 7 per cent or less in a steam locomotive. The steam locomotive is a sturdy machine conceived for reliable and ready use and thermal efficiency has not hitherto been of primary importance. With the increase in the price of coal and the availability of more and more oil in countries such as the United States of America, the diesel engine has come as a challenge to steam. The diesel locomotive is more efficient, has a better power/weight ratio, reliability, and has come

to stay in countries where oil is a serious competitor of coal.

The latest challenge to steam comes from the gas-turbine locomotive using powdered coal. Table I compares the steam, diesel-electric and gas-turbine locomotives under the heads of efficiency, fuel charges, maintenance charges, etc.

The efficiency of the coal-fired gas-turbine locomotive is 15 to 16 per cent as compared with 6 to 8 per cent of a modern coal-fired steam locomotive. In addition to higher efficiency, it has other important advantages over the steam locomotive : it has lower heat input per h.p./hr., less weight/power ratio, less maintenance, and less lubrication costs.

The drive of the coal-fired gas-turbine locomotive is through electricity generated by the turbine. A gas-turbine locomotive can, therefore, be converted into an electric locomotive if electric power is available.

The idea of a gas turbine is not new. There appears to be little doubt that the primitive rotary steam engines, such as Hero's aeolipile and Giovanni Branca's steam jet-operated turbine of 1629, inspired later inventors in their attempts to substitute combustion

\* Paper read at the Annual Meeting of the Indian Institute of Engineers held at Allahabad, January 1949.

**TABLE I\* — COMPARISON BETWEEN STEAM, DIESEL-ELECTRIC & GAS-TURBINE LOCOMOTIVES**

REF. LETTER	ITEM	STEAM (RECIPRO-CATING)	DIESEL ELECTRIC	GAS TURBINE
(a)	Approximate cost per horse power	£ 7	£ 17 10s.	£ 13
(b)	Efficiency at drawbar per cent	6-8	26-28	15-16
(c)	Mileage per year	180,000	250,000	Greater than 250,000
(d)	Time for tank filling and refueling	Greatest	Least	Small
(e)	High schedule speed	Lowest	Higher	Higher
(f)	Track wear	Large	Less	Least
(g)	Power braking	None	Full power	Full power
(h)	Approx. life in years	30	15-20	30
(i)	Maintenance	Lower	High	Least
(j)	Fuel costs (steam equal to 100), %	100	50-75	50-75
(k)	Lubrication costs as percentage of fuel costs	10	20-30	Less than 1
(l)	Water costs as percentage of fuel costs	10	Small	Nil
(m)	Starting effort	Minimum	Larger	Larger

\* From *Modern Gas Turbines* by Arthur W. Judge (Chapman & Hall Ltd., First Edition, p. 246) by kind permission of the author.

products for steam as a more convenient means of power generation by the turbine method.

The first patent on gas turbines was granted in 1791 in England to John Barber. John Barber may have originated the name "gas" turbine presumably because the fuel used in his combustion chamber was producer-gas. In his machine, producer-gas obtained from charcoal was burnt in a fire box with compressed air and the products of combustion made to issue against the blades of a turbine wheel. This and some other later designs failed to deliver any useful output of power. Fig. 1 explains the reason.

In a gas turbine, the turbine and the compressor (which feeds it) are on the same shaft. Consequently, the useful output of work from a gas turbine is the difference between the power developed by the turbine and the power absorbed by the compressor. The compressor absorbs about 75 per cent of the energy developed by the turbine. Hence, the efficiencies of the compressor and of the turbine are important factors. Fig. 1 shows the relation between the thermal efficiency of the gas-turbine unit, at different inlet temperatures, as a function of the product of the efficiencies of the compressor and the gas turbine. The thermal efficiency values are defined by the ratio  $W/H$ , where  $W$  is the

net-work output and  $H$  is the energy supplied by the fuel in the combustion chamber. It will be seen that the thermal efficiency of the gas turbine is zero at a gas inlet temperature of 1,000°F. when the overall efficiency (compressor  $\times$  turbine efficiency) is 53 per cent) and this efficiency rises only to about 5 per cent as the inlet gas temperature is increased from 1,000° to 1,500°F. Hence, to attain a reasonable efficiency of about 15 to 18 per cent at a gas temperature of 1,000°F. at turbine inlet, an overall efficiency of turbine and compressor of about 70 to 75 per cent is essential. With the increasing experience in the design and building of efficient compressors and turbines, the gas turbine for generating power has become a workable proposition. Other factors which have contributed to the rapid development of gas turbines are :

(1) The experience gained in the use of gas turbines in superchargers for diesel and aircraft engines.

(2) The experience gained in the use of gas turbines for the utilization of the energy of the exhaust gases in the working of processes like the Houdry oil-cracking process. In the Houdry process, large volumes of compressed air are utilized in burning carbon deposited on the catalyst and a gas turbine is used to recover power from the hot compressed gases.

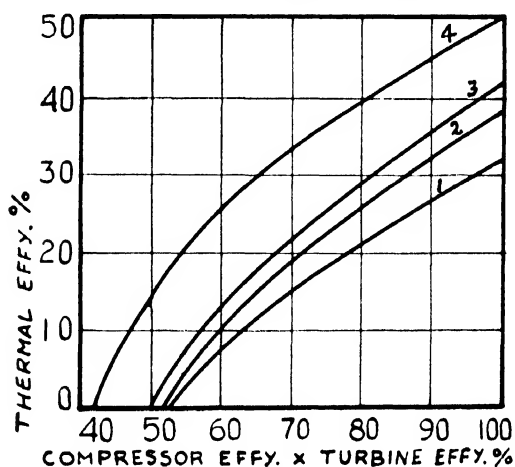


FIG. 1 — THERMAL EFFICIENCY OF A GAS TURBINE AS A FUNCTION OF THE OVERALL EFFICIENCY OF COMPRESSOR AND TURBINE FOR DIFFERENT TURBINE INLET TEMPERATURES (WITHOUT RECUPERATION OF EXHAUST HEAT). THERMAL EFFICIENCY AT COUPLING OF TURBINE AT (1) 1,000°F.; (2) at 1,200°F.; (3) AT 1,500°F.; AND (4) AT 2,200°F. (From *Modern Gas Turbines*, Chapman & Hall Ltd.)

(3) The experience gained in the use of gas turbines for the utilization of the energy of the exhaust gases from Velox steam generators. In the Velox boiler, the fuel, gaseous or liquid, is burnt with air in pressure combustors and a gas turbine is used to recover power from the exhaust gases.

(4) Research on the development of alloys to withstand the rotating stresses of the turbine blades operating at temperatures greater than 1,000°F.

(5) Fig. 2 shows the elementary gas-turbine open cycle.

Air is drawn and compressed by compressor (1), the fuel-air mixture burnt in combustor (2) and the hot gases are exhausted through gas turbine (3). The compressor and gas turbine are on the same shaft. *The difference between the power developed by gas turbine and that absorbed by the compressor is the output of useful power.*

Regeneration of heat from the exhaust gases, pre-heating of air before it goes into combustion chamber, multi-stage compression, multi-stage combustion, multi-stage exhaustion through the turbine and other factors contribute to the better efficiency of the gas-turbine cycle.

From the elements of the gas-turbine process outlined above, it will be seen that the output of power comes from the heat added to the gases by combustion of the fuel in the combustion chamber (2). The rate of heat release in modern gas-turbine combustors is of the order of 300,000 to 1,000,000 B.t.u./cu. ft./hr. as compared with a heat release of 20,000 to 40,000 B.t.u./cu. ft./hr. in the combustion chambers of large modern boilers fired with powdered coal. Such a high heat release rate can be obtained in a gas turbine because combustion is carried out under pressure. Naturally, liquid fuels are easy to handle and control in a gas-turbine combustor.

The use of powdered coal comes as a substitute for liquid fuels as coal is cheaper than

oil and is easily available. Considerable research has to be carried out, however, before the problems arising out of the use of powdered coal as a fuel for gas turbine are solved.

Fig. 3 is a flow sheet for a gas turbine using powdered coal at the rate of 500 lb./hr.

The flow sheet is based on a 400 per cent supply of excess air for a type of coal generally used for steam raising. The large excess of air is required to reduce the temperature of the products of combustion to 1,200°F. at the turbine inlet. The turbine inlet temperature is limited by the suitable metals and alloys to withstand (when hot) the high rotating stresses in the blades of the turbine. Further research on the development of better alloys to withstand the rotating stresses at still higher temperatures will improve the overall efficiency of the gas turbine.

The need for a large compressor for any preliminary work in gas turbines becomes evident from the study of the flow sheet. A compressor of one-eighth the capacity of the one mentioned in the flow sheet consumes about 75 kW./hr.

The main problems requiring detailed study in the powdered-coal gas-turbine development work are:

- (1) Crushing and grinding of the coal to the requisite fineness.
- (2) Conveying the powdered coal and feeding it under pressure into the combustor.
- (3) Combustion of powdered coal under pressure.
- (4) Removal of fly ash from the combustion products (at high temperature and pressure) before the gases pass to the turbine.
- (5) Co-ordination of all these operations so that the gas-turbine generator generates power under controlled conditions.

The last factor is specially important when the application of gas turbines for locomotive purposes is considered, as the load demands of locomotive engines vary widely and these variations have to be met quickly.

### 1. Fuel Handling & Grinding

Since the combustion chamber has to operate under pressure, the powdered coal has to be conveyed and fed into the combustion chamber against the operating pressure of the combustor. It is not economical to use an ordinary grinding mill modified for pressure grinding, as this would involve heavy and expensive equipment. A reasonable method would be to crush and grind in ordinary mills and transfer the

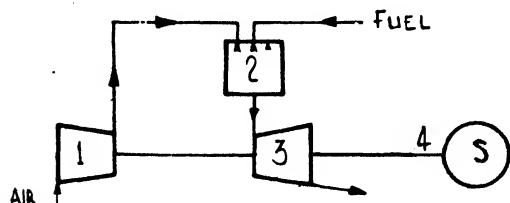


FIG. 2—ELEMENTARY GAS-TURBINE UNIT: (1) AIR COMPRESSOR; (2) COMBUSTOR; (3) GAS TURBINE; (4) GENERATOR.

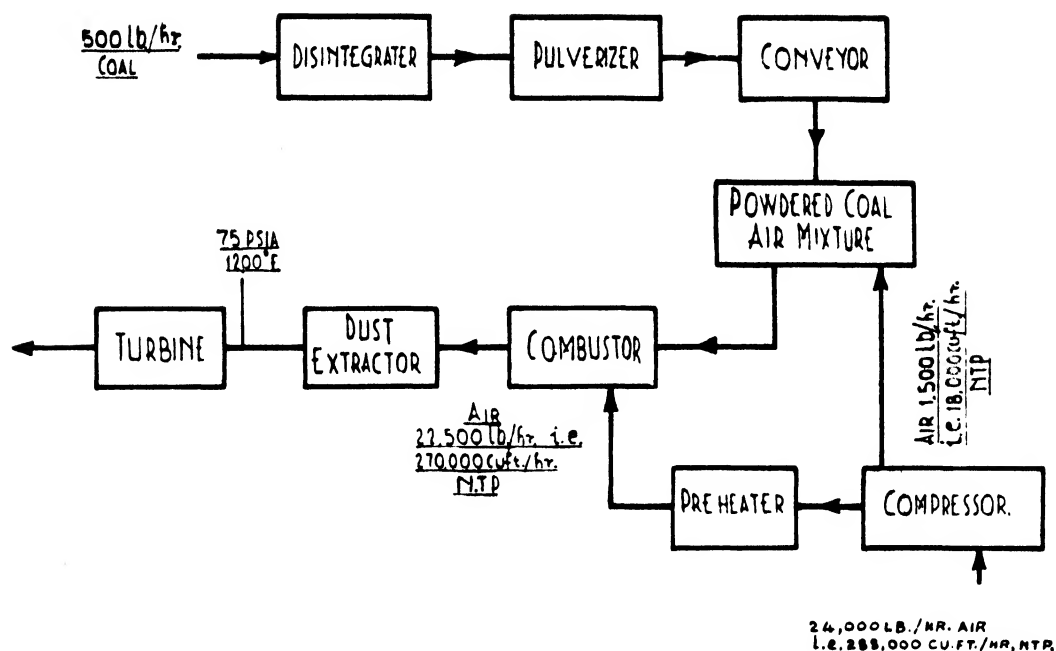


FIG. 3 — FLOW SHEET FOR A GAS TURBINE USING POWDERED COAL AT THE RATE OF 500 LB./HR.

powdered coal from atmosphere into pressure cycles. There are two methods of doing this: (1) by lock hoppers, one hopper being charged while the other under pressure feeds coal into the cycle; and (2) by feeding coal from a bunker at atmospheric pressure to a pressure bunker by a screw conveyor built in such a way that the thoroughly packed coal serves as a seal. In actual working it appears that the second method is not convenient, as the free and regular flow of powdered coal is obstructed by a thoroughly packed seal of finely powdered coal.

A method using the energy of compressed air for grinding and conveying would be most suitable under the circumstances. Such a method has been developed by Yellott and Singh<sup>1</sup> and their equipment, called "coal atomizer", is useful for crushing and conveying coal. The coal atomizer works on the principle of the "explosion" process of pulverizing materials. It has been used earlier by Dean and Gross<sup>2</sup> in the U.S. Bureau of Mines in their extensive investigations into the possibilities of crushing ore by the "explosion" process. The material to be pulverized is first subjected to a high pressure and the pressure is then released rapidly, the rapid release producing disintegration of the material. In actual practice, the rapid pressure release is obtained by the

use of a throat through which compressed air moving at 50 to 100 ft./sec. conveys the material to be pulverized. The preliminary crushing is done outside the atomizer, and the atomizer is used only for final pulverizing. The pulverized coal is carried into the combustion chamber by compressed air.

## 2. Combustion of Powdered Coal under Pressure

Very little quantitative data are available concerning the combustion of pulverized coal particles in suspension, and still less on the effect of pressure on the rate of combustion of powdered coal. It can be anticipated that the rate of combustion is much higher when the coal is in a pulverized condition and suspended in air in a pulverized form; the area of contact between coal and air is considerably increased and there is intimate contact between air and coal. Further, increased pressure will speed up the rate at which oxygen reaches the surface of the particle and thus shorten the time required for burning out. Experimental work carried out in the U.S.A. for the Locomotive Development Committee has shown that an increase in pressure results in a shorter flame.

The actual design of the combustion chamber depends primarily on how it is proposed to remove the ash and what is done

with the air required for dilution. At the high rate of heat release in the combustion chamber, it is obvious that the ash of all coals will pass through a molten phase. One method of dealing with molten ash is to delay the admixture of excess air and keep the temperature high. The molten ash is allowed to deposit on refractory-lined walls of the combustion chamber and/or thrown to the lower part of the combustion chamber where the slag can collect and be tapped off. The removal of slag from the combustion chamber has to be carried out under pressure. The alternative method of removing all the ash would be in a solid form. In this method the products of combustion are chilled, by the introduction of large excess air, to a temperature of 1,500°F. and then removed.

The walls of the combustion chamber, in the case of the first type of combustor, have to be designed to withstand the attack of slag at 2,700° to 3,100°F., whereas if the ash is removed in a solid form, the combustion chamber can be of metal with arrangements for rapid air-cooling of the walls.

The metal chamber will obviously be lighter than the slagging chamber and have greater flexibility: it is more suitable for mobile purposes, as in a gas-turbine locomotive. However, a slagging chamber would enable the removal of a large percentage of ash in molten condition, and this is of considerable help in the later cleaning of the products of combustion before they are fed to the turbine.

In both the types of combustion chamber, an intimate mixing of air and coal is obtained by giving a rotary motion to the particles and increased turbulence to the air.

The temperature to which the exhaust gases from a gas-turbine combustor can be raised is dependent on the percentage of excess air supplied for combustion. While it is desirable to increase this temperature to a high value for great efficiency, there is a limit beyond which the temperature must not be taken; this limit is set by the metals and alloys available for use in the highly stressed rotating elements (when hot) of the turbine. With the best existing alloys available, this temperature is restricted to a maximum of 1,200°F. for a reasonable operating life of a gas-turbine unit.

### 3. Removal of Ash from the Products of Combustion

Of primary importance to the powdered-coal gas turbine is the removal of ash from

the products of combustion before the hot gases are led through the turbine blades. Experience with Velox boilers operating on blast-furnace gases (containing 0.02 gm. per cu. ft.) has shown that particles below 5 $\mu$  are relatively harmless, but larger particles, particularly upwards of 40 $\mu$ , are very abrasive. A high efficiency of gas cleaning is needed in the range of this particle size.

The dust content of the flue gases from pulverized fuel-fired boilers is about 12 gm. per cu. ft. but in the coal-fired gas-turbine combustor, the concentration of dust may be smaller because of the large volume of excess air supplied.

The relatively high temperature of the gases and their enormous volume preclude the use of electrostatic precipitators. A satisfactory way of removing the dust, however, appears to be by the use of cyclones, in which the products of combustion containing the chilled ash particles are given a rotary motion. As a result of centrifugal forces, the heavier ash particles are thrown against the walls of the cyclone chamber and are removed through the bottom of the cyclone. The modern tendency in the design of cyclone separators is towards the use of multi-cyclone cells rather than a single cyclone; multi-cyclones are more efficient.

A multi-cyclone cell consists of several small cyclones, all arranged in parallel, with common dust-collecting hoppers. The gases to be cleaned are distributed through the different small cyclones of the multi-cyclone cell and are brought together into a central exit after cleaning.

It is of interest to compare the gas-cleaning equipment of a coal-fired gas turbine with that of a steam boiler operating on pulverized fuel. Assuming equal efficiencies, the gas-turbine dust extractor will have to remove about 25 per cent more ash (all the ash of the coal instead of 70 to 80 per cent of it), and will have to deal with 4 to 5 times the quantity of excess air. Further, the temperature of exhaust is higher (about 1,250°F.) as compared with a temperature of about 300°F. for a pulverized fuel boiler; this introduces a factor of 2.2 into the volumes handled. However, the higher pressure reduces the volume of the exhaust gases to about a quarter. The dust extraction of pulverized-coal gas turbine has thus to be of a higher capacity, roughly twice that for the pulverized fuel-fired boiler, and has to be designed to withstand considerably higher pressures and temperatures.

### Conclusions & Development Problems

Of the problems involved in the development of the powdered-coal gas turbine, some are of a fundamental nature and investigations can be made on a laboratory scale. Others have to be studied on a full-size or semi-size plant.

Investigations have to be done on :

(1) Pneumatic grinding, especially aiming at a reduction in the consumption of energy required.

(2) Pressure combustion of powdered coal. At higher pressures, the rates of heat release and heat transfer will be of an order quite different from those obtained in known solid fuel-fired appliances, and special studies have to be made of flow conditions in burners and combustion chambers.

(3) The behaviour of ash under varying load and combustion conditions and its removal.

Some of the above problems are of a fundamental and formidable nature, and much work will have to be carried out in special combustion chambers and, eventually, under actual operating conditions before success is achieved.

The author wishes to thank the Director and the Assistant Director of the Fuel Research Institute for helpful criticism and suggestions in the preparation of this paper.

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## Manufacturers' Industrial Exhibition

THE ALL-INDIA MANUFACTURERS' ORGANIZATION is sponsoring a "Manufacturers' Industrial Exhibition" in New Delhi during February 1950. A "Manufacturers' Exhibitions Society" has been formed for organizing and running the exhibition.

The Exhibition is open to all classes of goods of Indian manufacture, whether produced in factories or on a cottage-industry basis ; and also to foreign manufactures of the

following types of goods only, viz. capital and semi-capital goods and industrial, raw and processing materials including engineering stores.

Manufacturers who have not yet contacted the All-India Manufacturers' Organization's Central Office, Church Gate, Bombay, or its Delhi Provincial Board, opposite the Odeon Cinema, New Delhi, may do so.

# REVIEWS

**The Essential Oils, Vol. II—The Constituents of Essential Oils**, by Dr. Ernest Guenther—co-author Dr. Darrell Althausen (D. Van Nostrand Company Inc., New York ), 1949, pp. 852. Price \$ 10.

IT WAS WITH A THRILL OF REMINISCENT delight that I turned into my old files of *The Perfumery and Essential Oil Record* of London and re-read the six special lectures delivered in London during April and May 1912 on the chemistry and synthesis of aromatic chemicals used in perfumery and on the preparation of essential oils and their composition. Stalwarts like Sir William Tilden, F.R.S., W. H. Perkin, F.R.S., and Mr. John C. Umney ( Editor of the *Perfumery and Essential Oil Record* of London ) gave in their lectures a compendious survey of the then available knowledge on the subjects they handled. The audience was wonder-struck at the marvellous advances already made in the chemistry of essential oils and at the promise held out for the future of this special branch of organic chemistry by ceaseless research activities in England and on the continent of Europe and in America. This promise has been more than fulfilled! Yet, the work is still going on and will go on.

All the same, can anyone forget that all this wonderful progress occupies only a minor part in the vast field of organic chemistry? Dr. L. Ruzicka, in a notable statement, observes as follows:

"It is perhaps natural for a research worker specializing in a restricted field like the perfumes to over-estimate the importance of this small group of naturally occurring compounds in the vast domain of organic chemistry. It need scarcely be pointed out that the development in the chemistry of perfumes that has taken place in the last century would have been inconceivable were it not for the huge scientific structure of organic chemistry, incomplete though that edifice still is. It may be compared to a cathedral to which, even after centuries of labour, some work still remains to be done on small decorative details: Can a humble workman be blamed if he looks proudly up

at the building and finds its chief beauty to be the obscure handiwork, high on some remote tower, which represents the labours of his own company of artisans? . . . "

Dr. Guenther, in his second volume on *Essential Oils*, throws into bold relief the results of the labours of this company of artisans in a vivid manner that compels the admiration and respect of anybody that cares to have an intelligent view of the works. Further, all these have been chronicled in a planned and systematic manner. In a chapter devoted to the historic aspect of the subject in the first volume on *Essential Oils*, proper tributes have been paid to those pioneers who laid the true and enduring foundations of synthetic organic chemistry.

Necessarily, the second volume on *Essential Oils* can only be a book of compilation which will take the rank of a valuable reference book. In his preface to the book, Dr. Guenther says as follows:

"The present volume, the second in a series on the essential oils, represents an attempt to eliminate at least some of the tedious 'spade-work' necessarily performed by the essential oil chemist. To this end, data on several hundred of the most important natural constituents of essential oils have been assembled into one volume, in the form of monographs, and brought as nearly up-to-date as possible. The structural formulas, occurrence, methods of isolation and identification, physico-chemical properties of these compounds have been described. Succeeding volumes of this series will deal with individual essential oils and their chemical composition, and frequent reference will be made to the monographs contained in this volume.

"A secondary aim of the present work is to stimulate further research in the field, particularly on essential oils of which the chemical composition is, so far, either only partially elucidated or entirely unknown."

It thus becomes evident that we cannot expect to find a mention of synthetic substances that are used widely in perfumery but not naturally occurring in essential oils. For example, such well-known synthetics like musk-xylol, hydroxycitronellal, amyl-

cinnamaldehyde- $\alpha$ , etc., etc., will go unnoticed as falling outside the strict scope and the planning of the book. Dr. Guenther may consider these remarks.

The scheme adopted in the book is one of great convenience. The name of the substance is first mentioned with its molecular formula and molecular weight, together with its structural description by following the accepted nomenclature in organic chemistry. Then follows the occurrence of the substance, its isolation, its identification and its physical and chemical properties. Wherever possible, the synthetic preparation of the substance is mentioned with details. Then follows the use that the substance finds in arts and industries, specially in perfumery. Lastly, apart from references to original papers on the subject-matter, additional literature has been suggested for the benefit of individual workers who are particularly interested in the compound.

The first volume on *Essential Oils* contained a very valuable chapter on "The Examination and Analysis of Essential Oils, Synthetics and Isolates" by Edward E. Langenau. In a similar manner, in the second volume, a very useful and helpful chapter on "The Preparation of Derivatives of Essential Oil Constituents" has been written by Dr. Frances S. Sterrett.

While reviewing a monumental volume like the present one, it will look pedantic (nay, almost churlish) to be over-critical to select a minor omission or a minor discrepancy or a minor difference of opinion regarding the still unsettled constitution of a complicated organic compound. Dr. Guenther says in his preface:

"The task of writing this book has been a complicated one, requiring years of work — a result, among other things, of a confusion in older literature, particularly in regard to stereoisomeric compounds. The difficulties of compiling material for the volume were further increased by the fact that during the Second World War scientific literature from continental Europe, the U.S.S.R., Japan and other parts of the world was not available."

In a private letter to me, written in September 1949, Dr. Guenther states as follows:

"With the completion of the fifth volume, I will have spent 10 years on this work alone, not counting the many years of travelling and writing articles for various trade papers which described the results of my field work."

The third volume will be available to the public during November 1949 followed by the last two volumes during 1950.

Dr. Guenther has earned the gratitude of all scientists interested in this subject. Mere congratulations will look too formal.

Except during the short period that Dr. J. L. Simonsen (Now Sir J. L. Simonsen) was working in India, our country has not contributed much in the field of original researches on essential oils. India happens to be the home of the most prized sandalwood oil, indispensable in any good perfume. India also produces cinnamon-leaf oil, lemon-grass oil, palmarosa oil, eucalyptus oil, linaloe oil and turpentine oil. Yet, practically all the scientific work on these oils have originated from the laboratories of the western countries. At the present time, some sporadic attempts are being made to distil new essential oils from the aromatic plants grown in India and the usual physical and chemical constants like specific gravity, optical rotation, refractive index and ester values and finally the solubility in 70 per cent alcohol — only these are determined and published. We have yet to build up a school of research on essential oils in India. Probably, one of the drawbacks has been the lack of well-equipped organic chemistry laboratories with a good reference library and adequate grants for the purchase of special chemicals required in organic research. The facilities for this type of work at the Forest Research Institute at Dehra Dun and the Indian Institute of Science, Bangalore, happen to be fairly satisfactory and there are newer institutions that have come into existence like the National Chemical Laboratory, Poona. What is wanted seems to be a savant who is prepared to devote his life-long work in this specialized field and train a band of devoted workers under his guidance and inspiration.

We shall be looking forward to the next three volumes on *Essential Oils* by Dr. Guenther.

S. G. SASTRY

**Crystals and X-rays**, by K. Lonsdale (G. Bell & Sons Ltd., London), 1948, pp. viii + 199. Price 21s.

DR. LONSDALE HAS DEVOTED A LIFE-TIME to the study of the structure of crystals by X-rays, and the volume under review has, therefore, all the weight of her authority. While the book does not lay claim to being



the last word on X-ray crystallography or even as a text-book for advanced students of this subject, it will be most helpful to those who propose to take up these studies for the first time or who, while working on routine methods in X-ray crystallography, do not fully realize the implications of these methods. In about 200 pages Dr. Lonsdale has covered a very wide field and brings the reader up to a point where he may, with advantage, turn to advanced treatises or original papers on the various topics treated in the book.

Of special interest to the reader would be the historical introduction which gives an account of the early history of Laue's discovery and of later developments on crystallographic analysis and the application of these methods for the determination of the structures of gaseous and liquid states.

The descriptions of the procedures employed for the generation of X-rays, production of narrow X-ray beams, generation of rectified high voltage with various circuits and monochromatization of X-rays, which cover a whole chapter, would be found most interesting and instructive. Another chapter is devoted to a concise account of the elements of crystallography illustrated with excellent diagrams. Methods of crystal drawing, the principles of stereographic and gnomonic projections for crystal faces and the development of space groups are discussed in some detail. This is followed by a brief treatment of the X-ray determination of crystal geometry by stationary single crystal and powder photography, and more or less detailed methods for obtaining indices of the planes and the determination of space groups.

The reader is thus led on to the problem of the distribution of atoms and electrons in a unit cell. The general structure regarding the scattering of X-rays by light and heavy atoms are first described, followed by the resultant scattering effect due to all the atoms in a unit cell. The description of intensity formulae and the derivation of structure factors are clear and precise, and a number of examples are given to explain how structure factors are used to determine the actual structures. Brief accounts of alternative methods for structure determination, based on the theory of chemical bonds and on optical and magnetic measurements, are also given. To complete the determination of the structure factor, a short account of the method for obtaining the electron density distribution by Fourier synthesis is added.

There is considerable difference between the intensity of X-ray reflections as calculated for ideally perfect crystals or mosaic crystals and the crystals as they occur in nature. Factors which give rise to these divergences, viz. crystal texture, crystallite size, imperfection of structure and the thermal vibrations of atoms are discussed, and attention is drawn to the importance of such studies on account of their close relation to the elastic properties of solids, specially in the case of metals and alloys and fibrous materials.

The whole subject-matter of the book is extremely well presented and takes the reader to the main lines along which X-ray investigations are proceeding or are likely to be fruitful in future. The get-up is excellent and it is adequately illustrated with figures and plates. Dr. Lonsdale has placed a large circle of students and research workers under a debt of gratitude by writing such a readable and informative volume on X-rays. It will, doubtless, find a place in every science laboratory.

P.K.K.

**Organic Coatings in Theory and Practice**, by A. V. Blom (Elsevier Publishing Co. Ltd., London), 1949, pp. x + 344 + 91 illustrations. Price \$ 6.75.

THE RAPID DEVELOPMENT OF THE RESIN industry in the past few years has made available numerous materials with a wide range of properties that are of considerable interest for coating purposes. Today a resin can be tailor-made to the exact requirements of a certain physical and chemical behaviour and it remains for the paint and varnish technologist to process it to the best of advantages it offers. The book under review provides quite a useful link between the resin manufacturer and the paint technologist and enables each to understand and appreciate the wide scope of the subject.

The book, as the author himself admits, is by no means complete and the author has restricted himself to such topics as are of importance to both theory and practice of organic coatings. It provides a good review for a person seeking for a collected but concise and brief discussion of the subject. The author's vast experience in the line and information, which he collected from several authoritative sources, have made the text a very valuable guide to the man in the laboratory as well as in industry. He has

gathered in book form what one can only acquire by reference to a number of original papers.

After a brief introductory chapter on the historical, structural and other physical aspects of polymeric materials, the two following chapters are devoted to the chemistry and preparation of natural and synthetic organic film-forming materials. Although these two chapters constitute one-third of the book, the vastness of the subject has restricted the author to give only a brief account of these materials. A little more discussion, particularly on the synthetic materials, would have been quite useful. The value of the next two chapters on "Film Formation" would have been greater if they had been supplemented by a fairly detailed discussion on the important phenomenon of adhesion. Finally, the chapter on "Testing of Films" is quite thorough, illustrative and particularly well written. Reference to original papers and frequent illustrative diagrams are a very valuable feature of the entire book. Reliable theories are presented throughout the book in order to provide basis for further technical development. An additional chapter on the uses and applications of coatings giving the exact requirements for a particular job, type of equipment employed and advantages and disadvantages of each coating material should have been included to make it more valuable from a practical aspect.

In spite of these shortcomings, the author has certainly succeeded in making the book an interesting and informative reading.

S. L. KAPUR

**Fluid Dynamics**, by Victor L. Streeter (McGraw-Hill Book Company), 1948, pp. xi + 263. Price \$ 5.00.

IT IS A GREAT PLEASURE TO REVIEW THE book *Fluid Dynamics* by Victor L. Streeter. The book deals with the fundamental principles of ideal fluid in two and three dimensions and is written in a very clear and lucid style. The book is divided into 12 chapters. After describing, in chapter I, the conception of flow of fluid, the author passes on to develop basic mathematical theories in the II, III and IV chapters. In chapter V the conception of complex variables has been introduced and in chapter VI the problem of stream lines, equipotential lines, etc., have been well treated from the standpoint of conformal transformation. In chapter VII examples requiring multiple transformations are worked

out leading to irrotational flow around in airfoil with circulation. The chapter VIII deals elegantly with the Schwarz-Christoffel theorem in relation to free stream-line transformations. The chapter IX on vortex motion is very well planned and interesting and deals with the vortex motion where the vorticity is confined to lines, tubes and sheet or is unconfined and examples are considered for both two-dimensional and three-dimensional flow. In chapter X, the equations of viscous flow, as given by Navier and Stokes, have been developed and solved for a number of interesting cases in the next chapter, such as the theory of lubrication, the steady flow through circular tubes, Stoke's law, Reynolds number, etc. In the last chapter, the Navier-Stoke's equations are applied for laminar boundary layer, i.e. when the motion of the fluid particles in the boundary layer are sensibly in a straight line.

The book would have been very suitable for the courses of Mathematics and Mathematical Physics in most of the Indian universities if it had included one chapter describing the motion of solids through liquids and another on the theory of waves in general.

The characteristic of the book is that the underlined principles of fluid mechanics have been logically and clearly explained supported in each case by solving some interesting examples to illustrate the principle. A number of numerical questions have been added at the end of each chapter although the number of examples to be solved by the students are not adequate.

The book will be found extremely useful by the students who desire to get a clear grasp and a thorough understanding of the fundamentals in fluid dynamics and is worth keeping in every library. The printing and get-up of the book are excellent.

R.C.M.

**Physics and Chemistry of Cellulose with particular reference to Rayon**, by P. H. Hermans (Elsevier's Polymer Series, Vol. 2) (Elsevier Publishing Co. Inc.), pp. xxii + 534 with 244 illustrations. Price 50s. net.

THIS IS AN AUTHORITATIVE MONOGRAPH which will be valued by those interested in cellulose research and by rayon technologists. Many of the investigations described in the book have been carried out in the Institute for Cellulose Research of which Dr. Hermans is the Director. It is remarkable that an

organization of the relatively small rayon industry in Holland should have sponsored an extensive programme of fundamental research on cellulose.

The book is divided into three parts. Part I gives a critical survey of the literature on crystalline and micellar structure, swelling and dissolution, degree of polymerization and its determination, chemical reactions, and structural variations in natural and regenerated celluloses. Part II is a detailed treatment of the work of the author and his school on the fine structure of cellulose fibres, using known methods with higher precision as well as new technique. Most of the material has already appeared in an earlier monograph of the author (*Contributions to the Physics of Cellulose*, Elsevier, 1946). The subjects treated in this part are morphology of natural and regenerated celluloses with special emphasis on the occurrence of skin and core regions in the latter; the sorption of, and swelling in, water and their dependence on the proportion of amorphous regions in the fibre. In discussing the dual function of hydroxyl groups in cellulose chains (hydrogen bond formation and hydrophilic action leading to swelling in water), the possibility of hydrogen bond formation in the amorphous regions appears to have been overlooked. The decrease in the swelling of viscose rayons caused by increase in orientation, mainly in the amorphous regions, indicates the formation of more and more hydrogen bonds in these regions. It is well known that crystallinity does not alter to any appreciable extent during this increase in orientation. The chapter on density measurements sets out the author's experimental data. The conventional view that water in fibres is under very high pressure, as an explanation of the difference in the observed density of cellulose in inert media and in water, is rejected in favour of a theory based on a concept of the packing of "spheres" of glucosidic groups and water molecules in the amorphous regions. The evaluation of

orientation by measurements of birefringence is described, and theoretical limits of birefringence in native and regenerated celluloses are deduced. After discussing X-ray spectra of fibres, there is a full account of the mechanical properties and the fundamental issues in stress-strain tests are analysed. While interest in earlier days was mainly centred round the crystallites or micelles in the fibre, the present trend is to devote more attention to the amorphous regions, in view of their relation to useful fibre properties such as dye absorption, extensibility and flexibility. A recently developed technique of Hermans for evaluating the crystalline-amorphous ratio in cellulose is described fully.

Part III is mainly devoted to the researches of the author on the "model fibres" (isotropic regenerated cellulose monofilaments with circular cross-sections). Work on the deformation of such model fibres swollen in various states has thrown fresh light on the problem of the arrangement of chain molecules on gelation and on subsequent application of force in the direction of the fibre axis. The net-work structure of Kratky and W. and H. Kuhn's concept of chain molecules kinked at random and statistical chain element are examined and fitted into the general picture.

The translation is faulty and there are unusual expressions such as conception (concept), artificial cellulose (regenerated cellulose), gelatination (gelation), aceretion (accessibility), asseveration (categorical statement), and signification (significance). Names of Indian and other authors are spelt erratically, e.g. Banjeri and Ghos (Banerji and Ghosh), Chawdhury and Bhardaw (Choudhury and Bardhan). The book is an advanced and highly specialized treatise, which is not easy to read, and is primarily intended for investigators in the field of the fine structure of fibres. Although the title is "Physics and Chemistry of Cellulose", the chemical aspects receive but scant attention.

K.V.

# NOTES & NEWS

## Anhydrous Ammonia as Fertilizer

DEVELOPMENTS IN THE APPLICATION of anhydrous ammonia as a nitrogenous fertilizer and its utilization are described (*Chemie et Industrie*, 1949, 62, 26).

Experiments conducted in the United States have demonstrated in the case of cotton, wheat and oats that the soils treated with ammonia gave the same yield as with nitrogenous fertilizers. Subsequently, tests were carried out under the auspices of the *Comptoir Francais De l'Azote*. An ammonia reservoir mounted on a tractor injected ammonia into the soil under controlled conditions. The treatment was found to give ideal results at the sowing time because then the ammoniacal nitrogen is readily assimilated by the roots. The depth of application of ammonia in the soil depended on the season of application and varied from crop to crop. Nitrification was related intimately to the acidity of the soil and the line of application of ammonia to the soil. A soil with a pH lower than 5.5 had to be treated in autumn because in winter there is practically no nitrification and ammonia remains in the soil as ammoniacal nitrogen up to spring. In soils with a pH higher than 6 nitrification is appreciable and the nitrate nitrogen is liable to be washed away by the rains.

Substitution of nitrogenous fertilizers by anhydrous ammonia saves expense in transport and possibly also in the consumption of energy. For obtaining 1 ton of fixed nitrogen in the form of anhydrous ammonia, the expenditure of energy is of the order of 4 tons of carbon. Its transformation into solid nitrogenous fertilizers demands these quantities of carbon for every ton of fixed nitrogen: 0.25 tons for synthetic sulphate; 1 ton for ammonium nitrate; 2.5 tons for sulphate by the gypsum process; 3.3 tons for calcium nitrate; and 7.5 tons for sodium nitrate.

## Caking of Ammonium Nitrate

METHODS FOR OVERCOMING THE caking tendency of ammonium

nitrate have been reviewed (*Industr. Chemist*, 1949, 25, 399).

Various physical phenomena causing caking of the salt and factors influencing the quality of the cake formed, e.g. moisture content, transition temperature, particle size, duration and method of storage, etc., have been dealt with.

The methods adopted so far are:

(1) Coating the salt with some organic grease or oil or solid in order to diminish its absorptive capacity for moisture from the atmosphere. The coating of ammonium nitrate with organic materials has two grave drawbacks. It is impossible completely to cover the granules of the salt so that the results obtained are generally disappointing and the quantity of organic addition necessary for worthwhile results, usually about 1.0 per cent, is such that the explosive decomposition of ammonium nitrate is considerably sensitized thereby and a serious explosion hazard in the treated salt results. (2) Dusting the salt to aid the free running properties of the salt and minimize the setting tendency. Most inorganic dusting agents have the effect of desensitizing explosive compositions containing ammonium nitrate, since they are rarely very effective in proportion less than 1 per cent of the salt; thus the process is of limited applicability in the explosives industry. The only dusting agents for ammonium nitrate likely to be free from objection for use in the explosives industry are expensive, finely divided, bulky and insoluble, which maintain their identity when mixed with ammonium nitrate. A few complex silicates and some sulphates, oxides and phosphates of the insoluble salts are likely to be useful. Precipitated calcium phosphate is fairly efficient as an anti-setting agent up to a moisture content of 0.5-0.6 per cent and is recommended. In the production of fertilizers, however, suitable additions of dusting agents in proportion larger than 1 per cent of the salt can be considered and the dusting of coarse-grade ammonium nitrate with about 5-10 per cent of some inert addition is recommended as an

effective method of making possible the handling of ammonium nitrate as a fertilizer, without increasing its detonation risk or decreasing its nitrogen content below about 32.5 per cent.

Microscopic study of coloured dusting agents, e.g. pigments distributed on dry ammonium nitrate, showed that the coating effect on a relatively dry salt is very small. To obtain better coatings, a few water-soluble substances in aqueous solution have been suggested. Other methods include moistening the surface of the granules with phosphoric acid or sulphuric acid, exposing to gaseous ammonia, or applying pairs of substances in solution to give such insoluble compounds as ferric hydroxide, colloidal phosphates or ferricyanides.

A study of crystal modificants for ammonium nitrate has recently revealed a new method of great promise for reducing the setting tendency of ammonium nitrate, particularly for use in the explosives industry. A number of sulphonated organic dyestuffs have been found to modify the crystal structure of the ordinary temperature stable modification IV of ammonium nitrate from (110) acicular of prismatic forms to (010) plates, when the salt is crystallized in the presence of 0.10 per cent of the dyestuff. The most marked alteration is brought about by the acid triphenylmethane dyestuff acid magenta. Crystallization below 32°C. of a saturated solution of ammonium nitrate containing 0.10 per cent of this dye results in the deposition of extremely soft lamellar aggregates of the modified crystals, which, when isolated, are very fragile, thin plates or "scales".

It has been found that, if crystals of ammonium nitrate are mixed with an aqueous solution of acid magenta and dried so that a surface coating of about 0.03 per cent of the dye is applied to the salt, the setting tendency of the salt is very greatly reduced. The action of acid magenta is extremely effective even at moisture contents above 1.0 per cent.

This modification of ammonium nitrate by dyes is apparently confined to the modification IV, thus the efficacy of the anti-setting action of acid magenta is greatest at temperatures at which this modification of the salt is stable. The setting tendency of ammonium nitrate III, however, is somewhat reduced by the presence

of acid magenta, this possibly being due to a tendency of the salt to undergo habit modification, or the mere presence of the larger dye molecules on the surface of the salt granules.

Acid magenta treatment of ammonium nitrate has the following advantages as compared with the earlier methods of reducing the setting tendency: (a) the quantity of the partly organic addition is insufficient significantly to increase the explosiveness of the salt, or to reduce the sensitiveness of explosive compositions; (b) the anti-setting effect is maintained even when the treated salt is incorporated in gelatinous explosive compositions; (c) the dye neither affects the "heat test" of explosives, nor does it react with the salt in high temperature storage tests; (d) it is likely that use of acid magenta will allow a considerable extension of the range of particle sizes which can be handled without undue trouble from caking.

### Chromatography of Amino Acids

A SIMPLE MEANS OF ESTIMATING lysine and some of the other amino acids by chromatography of their copper salts of the acids is described (*Science*, 1949, 110, 163).

The salts were prepared by *Pope and Stevens* method as modified by the authors (*J. Biol. Chem.*, 1944, 153, 583). 0.02 c.c. aliquots of the solution was partitioned on paper strips in an aqueous phenol atmosphere for 6 hr. After drying in air, the strips were developed by painting with a freshly prepared 10 per cent solution of aqueous iron ferrocyanide. The characteristic pink colour of copper ferrocyanide, which appears distinctly on drying, indicates the position and relative amounts of various amino acids on the strip. Tests with 60  $\gamma$  of purified specimens showed glutamic and aspartic acid to have overlapping  $R_f$  values in the area 0.08-0.10. Lysine occurred in the region 0.42-0.50. Arginine and histidine overlapped in the 0.55-0.62  $R_f$  zone. The other amino acids, glycine, serine, threonine, valine, leucine, isoleucine, tyrosine, proline, hydroxyproline were found in the zone 0.7-1.0. Tryptophane, methionine, cystine and phenylalanine could not be identified by this technique, since they are oxidized during partitioning.

The band spread of the copper lysine salt is roughly proportional to the concentration in the range

30-60  $\gamma$  of lysine. With protein hydrolysates containing initially 0.5 mg.N/c.c. it was found that zein gave negative tests; wheat gluten gave faintly positive tests; and casein, gelatine, lactalbumin, human haemoglobin and fibrin gave bands which approximated roughly the lysine contents of the preparations.

The method is useful for the determination of lysine in urine specimens. Blood filtrates, however, could not be manipulated satisfactorily to give positive results.

Within limits this method is a useful one, but it cannot be expected to yield better than semi-quantitative results.

### Use of Detergents in Fat Determination

THE DETERMINATION OF FAT content by usual procedures involves in most instances the use of long and difficult methods and extensive laboratory equipment. A new principle—formation of a protein-detergent complex to break up emulsions and liberate the fat obviates these difficulties. This test, used for the determination of fat in milk, shows promise as a universal procedure for fat estimation (*Science*, 1949, 40, 121).

The materials used are: (a) a saturated solution of a fat dye prepared by mixing 500 mg. of Oil Red O in 100 c.c. of isopropyl alcohol and 2.5 c.c. of the clear solution added to a mixture of 100 gm. of a standardized non-ionic detergent, poloxyethylene sorbitan monolaurate, and 65 c.c. of 95 per cent ethyl alcohol and the new mixture shaken well; (b) standardized anionic detergent, dioctyl sodium phosphate.

A well-mixed sample (17.6 c.c.) of milk is placed in a Babcock milk bottle and shaken intimately with 7 c.c. of solution (a); 13 c.c. of reagent (b) is added without further shaking so that this last addition forms a layer at the bottom of the container which is then immersed in a water bath at 180°F. The water level in the bath should be approximately equal to that in the bottle. After 5 min. in the bath, water at 180°F. is added to the Babcock flask until the fluid level reaches the top of the graduated portion. The flask is then set aside at room temperature for 10 min., after which the percentage of fat (as in the Babcock test) is read by subtracting the lower meniscus reading from that of the upper meniscus.

100 duplicate samples of milk were tested for quantitative fat

content. The readings obtained by this method were the same as those resulting from the Babcock test.

Dioctyl sodium phosphate is an anionic compound in which the hydrophilic portion is negatively charged. It has a long chain of carbon atoms with a strong polar group located near the centre of the carbon chain. Mixing this detergent with milk and heating effects a dispersion of the protein layer around the fat globule, and liberating the fat so that it can combine with other fat molecules. However, the separation is not complete. When a quantity of the strongly hydrophilic, non-ionic detergent is added to the mixture, a clear solution and complete separation results.

### Separation of Organic & Inorganic Materials by Flotation

THE POSSIBILITY OF EMPLOYING flotation technique in the beneficiation and separation of inorganic and organic materials soluble in water is indicated (*Chemie et Industrie*, 1949, 62, 135).

The normal conditions controlling the flotability of ores are valid in this system also but the solubility of the inorganic materials in water creates complications. Pure water used in the flotation of ores is, therefore, replaced in this case by a saturated solution of salts taking part in flotation. The aptitude of the body to be wetted changes, and viscosity, modified by the dissolved salts, exerts an influence on the quality of froth produced. Tests covering mixtures of the salts of alkalis and alkaline earths, chlorides, nitrates, nitrites, sulphates and carbonates as well as influence of fineness of the powder, solubility of salts and temperature on separation of salts by flotation are discussed. Results of studies on the following are also given: (1) influence of flotation reagent (sodium oleate) for mixtures of  $\text{NH}_4\text{Cl}$  and  $\text{NaNO}_3$ ,  $\text{KCl}$  and  $\text{NaCl}$ , and  $\text{KNO}_3$  and  $\text{NaNO}_3$ ; (2) separation of salt mixtures with the same cation; (3) separation of mixtures of alkali salts and alkaline earths; (4) separation of salts of alkaline earths.

These experiments established a correlation between the speed of wettability and flotability though the relationship was not amenable to mathematical treatment.

The problem of separating organic materials by flotation is quite different owing to the hydrophilic character of most of the

solid organic compounds. The aim in this case is to augment the nature of one of the two compounds in the mixture by the use of a specific reagent. Not much success has been achieved in the case of substances of pronounced hydrophobic character, but satisfactory results were obtained when attempts were made to induce hydrophylic character of a differential nature in a specific manner.

K. VYASULU

### Magnetic Method of Extracting Gold

A MAGNETIC METHOD FOR SEPARATING gold from hitherto unworkable low-grade ores is described (*Sci. Newsletter*, 1949, 56, 153).

The gold in cyanide solution is adsorbed on activated carbon which is rendered magnetic, either through the incorporation of ground magnetite or by impregnation with an iron salt which is subsequently reduced, leaving pure iron in the pores. After the carbon has adsorbed the dissolved gold, a magnetic separation is effected. With larger carbon particles losses are correspondingly reduced. The method obviates the appreciable losses entailed by flotation treatment of low-grade ores.

### New Test for Free Chlorine or Bromine

THE *ortho*-TOLUIDINE TECHNIQUE and the benzidine blue and iodide replacement methods used for measuring minute traces of free chlorine, occurring in drinking water after sterilization, do not measure free chlorine but indicate oxidative potential, and results so given are, therefore, subject to considerable error, should oxidizing agents other than chlorine be present. A new test is described which is specific for free chlorine or free bromine (*Nature*, 1949, 164, 448).

The test is based on the fact that free chlorine reacts directly with cyanides to form cyanogen chloride which is allowed to react with pyridine (or a pyridine derivative) to form a quaternary compound. This, on subsequent condensation with aromatic amines, produces a ring fissure and the formation of intensely coloured di-anil derivatives.

The two reagents employed are made as follows: (a) sodium cyanide solution 1 per cent and (b) pyridine 25 per cent containing 2 per cent of benzidine hydrochloride.

5 c.c. of water is treated with 1 c.c. of cyanide solution to which is then added 5 c.c. of the pyridine reagent. The reddish colour which is fully developed within 10 min. may be compared visually against standards or measured in a photo-electric instrument.

The test is approximately twice as sensitive as with the toluidine reagent and, as stated above, is specific, except in the presence of free bromine.

### Mounting of Bacteria for Electron Microscopy

A NEW METHOD FOR MOUNTING bacteria for examination of their developmental changes under the electron microscope without interference with their normal mode of growth is described (*Nature*, 1949, 164, 352).

The method consists in growing the organisms directly on collodion-covered specimen grids, placed in firm contact with solid media or floated on the surface of a fluid medium, which are then transferred directly to the electron microscope, after fixation and sterilization. The metal specimen grids are covered in the usual way, preferably sterilized with ultraviolet light or in an oven. They are placed in the growth medium and each grid is inoculated in the usual way with a minute droplet of a suspension containing the organism. After appropriate intervals of time, grids are removed in turn, fixed by exposure to osmium tetroxide vapour, and sterilized by formalin vapour or in an oven. If disturbing quantities of the medium remain adhering to the grid, it may be floated off on distilled water for a short time after removal from the medium. Alternatively, good results on fluid media have been obtained by interposing filter paper between grid and growth medium. After sterilization, the grid is dried and mounted in the specimen-holder in the usual way.

The method permits examination of virulent and pathogenic bacteria after any length of time and with minimum of risk as compared with previous methods. Good results are obtained whether the collodion film or the metal grid is in contact with the medium. Detailed investigation, however, of the possible influence on growth of the grid metal is considered desirable. So far copper and nickel grids have been used; it may be preferable to employ a noble metal.

### Aluminum Condenser & Heat-exchanger Tubes

THE CORROSION RESISTANT PROPERTIES of tubes drawn from aluminum alloys MI and MI Clad which are covered by specification B 234-48T of the *American Society for Testing Materials* are evaluated (*ASTM Bull.*, 1949, No. 159, 44).

The alloys differ principally in that MI Clad tubes are more resistant to perforation under certain corrosion conditions. For most applications, MI tubes are adequately resistant to corrosion, but when salts of heavy metals such as copper, tin, nickel or lead are present, even in comparatively small amounts, the contaminated water may induce severe pitting or perforation of the tubes. Under such conditions, the use of MI Clad tubes is recommended.

The superior resistance of MI-clad tubes is the result of an alclad coating on the inside surface of the tube. The coating comprises about 10 per cent of the wall thickness and is of an alloy containing about 1 per cent zinc, which is anodic to the MI alloy (1.3 per cent manganese, balance aluminum) in most waters. The coating alloy is applied during the initial stages of manufacture and, in the finished tube, is integrally bounded to the MI "core".

This coating alloy is not, however, completely immune to attack and in course of time develops pits which penetrate to the bond between the "core" and coating. Electrolytic action then prevents corrosion of the "core" and perforation of the tube, at least until the area of the pit has increased so greatly as to reduce the electrolytic action because of the increased resistance in the electrolyte.

When the operating temperature range is not too great, the aluminum alloy tubes can be used in conjunction with steel tube sheets without danger of working loose. Corrosion of the ends of the tubes, because of contact with the steel, is generally negligible unless the chloride content of the cooling water exceeds about 50 ppm. If brackish or sea water is used, tube sheets of a suitable aluminum alloy should be used. Tube sheets of copper-base alloys must always be avoided.

Aluminum-alloy condenser and heat-exchanger tubes have been used successfully in the production of acetic and other aliphatic acids, acetaldehyde, naval stores, vegetable oils, formaldehyde, hydrocyanic acid, in the ammonia

recovery steps of the soda-ash process, in condensing sweet and sour hydrocarbon gases and liquids, in furfural and other types of lubricating oil-solvent extraction processes, and in condensing steam. They have been used in air and water-cooled atmospheric heat exchangers, air and natural gas compressor inter-coolers and after-coolers, lubricating oil and jacket water coolers, and numerous other types of heat exchangers.

### **Passivation of Enamel Surfaces**

SOME IMPORTANT RESULTS OF experimental acid pre-treatment of porcelain enamels on the acid resistance and abrasion resistance of treated specimens have been obtained at the U.S. National Bureau of Standards' laboratory (*Chem. Age*, 1949, **61**, 217). Tests made earlier to compare the relative effects of hydrochloric, acetic and citric acids on a number of enamels revealed that treatment with acetic acid, which produced only minor visible attack, strongly inhibited further attack when subsequently treated with citric acid, although the citric acid severely attacked the untreated areas of the same enamelled specimens. It was found that pre-treatment with acetic acid in concentrations from 0.5 to 50 per cent for periods of 5 min. or longer were effective against subsequent treatment with 10 per cent citric acid. Treatment with butyric acid for 15 min. produced the same effect, but neither tartaric nor lactic acid was effective. This passivation may be explained on the hypothesis that the acetic and butyric acids preferentially leach alkalis from the enamel surface, leaving behind a silica-rich film which is resistant to further solution in citric or stronger acids. With citric acid, on the other hand, the attack apparently proceeds to a greater depth before sufficient thickness of this film is built up to inhibit further solution. Treatment for 15 min. in 10 per cent citric acid appreciably reduces the abrasion resistance of certain enamels having Class B or Class A acid resistance (Porcelain Enamel Institute standard test), this effect being more pronounced on titanium-type enamels than on antimony-type enamels. In no case was the abrasion resistance of Class A enamels affected.

These two effects are related; the silica-rich layer resulting from preferential solution of the alkali is less resistant to abrasion than

the original surface, although more resistant to acid attack. The results suggest that titanium-type enamels are especially sensitive to the observed effects, and that routine inspection tests should be made in regular production to assure that the composition and processing of the enamels are maintained at optimum conditions.

### **Disintegration of Metals**

TECHNIQUES FOR STUDYING the disintegration of metals and the wear of metal bearings have been developed at the Commonwealth Scientific & Industrial Research Organization of Australia.

By attaching a tracer element (radioactive atom of the metal under test) to the molecules of the metal, it has been found possible to study the movement of molecules at extremely low temperatures.

The discovery has made possible the protection of tin cases against *tin pest* (disintegration of tin at low temperatures) by determining the exact degree of admixture of other metals required in tin to stabilize its molecules. Similarly, the amount of wear that takes place in a metal bearing can be accurately and quickly measured. One of the practical results of this is that it will now be possible to decide which is the best lubricant for a particular engine bearing.

### **Magnesium Alloy Systems**

MECHANICAL PROPERTIES OF SOME 200 alloys in 40 magnesium alloy systems are surveyed in a recent report released by the U.S. Office of Technical Services.

A preliminary investigation of warm-rolling procedures for dilute magnesium-base alloys indicated the possibility of increased ductility and roughness with moderate strength properties through these methods, although the data were too limited in scope to draw specific conclusions.

Further study of the magnesium-zinc-aluminum-tin system agreed with earlier results indicating that magnesium-5 zinc-3 aluminum-3 tin was the best combination of this particular system.

The promising magnesium-5 zinc-1 silver-1.5 manganese-0.3 calcium alloy still has the drawbacks of hot-shortness (tendency to break under stress when being handled under high temperature) and extreme sensitivity of its mechanical properties to small variations in composition and handling procedures.

The report suggests that magnesium-zinc-silver-manganese-calcium is the most promising composition for further study and development (*Chem. Age*, 1949, **61**, 318).

Copies of the report No. PB-97657, *New Magnesium Alloys* (243 pp.) can be had as a photostat costing \$ 31.25, or as a microfilm at \$ 9.00, from the Library of Congress, Photo-duplication Service, Publication Board Project, Washington 25, D.C.

### **New Alloy Steel**

THE DEVELOPMENT OF A NEW alloy steel capable of meeting the stringent engineering requirements at temperatures as low as  $-423^{\circ}\text{F}$ . has been reported by the Lebanon Steel Foundry, U.S.A. (*Chem. Age*, 1949, **61**, 214).

An austenitic cast ferrous alloy containing 19.50 per cent chromium and 9 per cent nickel, among other alloying constituents, the new alloy promises to have particularly useful applications in meeting the requirements of the chemical and some other processing industries. It has already been used effectively in the production of steel castings for pressure equipment in storage facilities for liquid oxygen used as a propellant for rocket engines. Such storage is maintained under high pressures at temperatures of approximately  $-298^{\circ}\text{F}$ . In prospect are other applications in various restricted ordnance fields.

The new cast steel has shown no structural changes down to  $-423^{\circ}\text{F}$ . and extensive tests have indicated appreciable increase in hardness directly proportional to the decrease in temperature. An added factor of engineering importance to designers and development engineers is seen in the fact that tensile strength of the particular cast austenitic alloy varies inversely with temperature, and in direct relationship with hardness. Ductility is lowered somewhat, however, but not in proportion to the increase in tensile strength, which is estimated to be approximately 200,000 p.s.i. at  $-423^{\circ}\text{F}$ ., with retention of adequate strength factors and resistance to embrittlement.

The alloy may be welded by either gas or electric processes without the need for subsequent heat treatment, since low temperatures do not necessarily impair the as-welded impact resistance. No embrittlement from low temperature has been noted in the cast alloy from welds or adjacent fusion



zones made from the same composition, nor any transition zone has been noted in test work covering a large number of production heats.

### Low-octane Gasoline as Motor Fuel

LOW-OCTANE GASOLINE, FORTIFIED by periodic injections of alcohol and water has given successful runs in tests conducted by the U.S. Department of Agriculture (*J. Franklin Inst.*, 1949, **248**, 190). The trial truck operated as well on 58-octane gasoline and alcohol-water injection as it would on regular gasoline, which has an octane rating of 74 or better. The alcohol-water mixture (85 per cent alcohol, 15 per cent water) was injected automatically into the truck's carburettor when the engine was under heavy load, as in climbing hills, accelerating in traffic, or passing other vehicles. This enabled the engine to give knock-free performance even with the low-octane gasoline.

In a second trial the engine of the truck was equipped with a high compression head (8.25 to 1 compression ratio). This made it necessary to supply the engine with fuel having an effective octane rating of at least 85. On this trip 74-octane gasoline plus alcohol-water injection was used satisfactorily.

Alcohol is equivalent to 100-octane fuel, and it can be used to increase the effective octane rating — or anti-knock quality — of gasoline. It thus makes possible wider use of low-octane fuels, which are cheaper and can be produced from petroleum in greater quantity than high-octane gasoline.

### Protective Clothing

COMPLETE RESISTANCE TO MOST acids and alkalis, light weight, and flexibility are some of the outstanding advantages offered by the protective clothing recently developed by *Plysu Products Limited* (*Industr. Chemist*, 1949, **25**, 399). The protective clothing has already been widely used in chemical laboratories.

The clothing is made from vinyl sheeting based on a vinyl chloride acetate copolymer resin which enables a coat to be manufactured  $\frac{1}{1000}$  in. thick where  $\frac{1}{10000}$  in. is the safe minimum, using sheets based on other synthetic resins.

This clothing offers complete resistance to acids. It is not affected by alcohol, steam, carbolic acid, chlorine or other corrosive

agents and it possesses good flame resistance because vinyl sheeting does not support combustion. No stitching is employed in the manufacture of this clothing, and all seams are heat sealed by a high-frequency welding process.

### New Vitamin Factor

A NEW VITAMIN FACTOR, BIOCYTIN, has been isolated from yeast. Biocytin occurs in nature only in infinitesimal amounts. Yeast extract has one part of biocytin per million of dry matter. More than 8 tons of the extract processed yielded less than  $\frac{1}{16}$ th oz. of the pure material (*Sci. Newsletter*, 1949, **56**, 137).

### Field Station for Ionospheric Investigations

IN RECOGNITION OF THE RESEARCH work carried out by the University of Calcutta in the ionospheric field, and to assist it in its present investigations, the Australian Council for Scientific & Industrial Research has loaned ionospheric apparatus (made in Australia) to the University.

The apparatus will be installed at Harinaghata, about 30 miles from Calcutta. The Council of Scientific & Industrial Research, India, has made a grant of Rs. 80,000 for the construction of the field station. Calcutta University, in collaboration with the Indian C.S.I.R., will train about a dozen students in the technique of ionospheric investigation each year.

### Statistical Training Centre

AN INTERNATIONAL TRAINING CENTRE giving courses on censuses and statistics in different parts of the world has been started in New Delhi.

The centre has been jointly sponsored by the United Nations Organization, the FAO and the Government of India for the purpose of helping countries in censuses and statistical techniques and in disseminating information on the international recommendations for the agricultural and population censuses to be taken in or around 1950.

### Cultivation of Drug-bearing Plants

A BOTANICAL EXPERIMENT STATION for cultivation of drug-bearing plants has been set up at the College of Ozarks, Clarksville, in the United States, to aid researches for establishing the best

conditions for growing the plants as a regular crop. Hydroponic installations are set up where 15-20 simulated conditions can be studied for each type of plant whose complete life cycle from germination to harvest is observed (*Ind. & Eng. Chem.*, 1949, **41**, 7A).

### Indian Standards

*Storage Batteries & Cells* — The Sectional Committee on Electrical Accessories and Batteries has prepared a draft Indian Standard Specification for Lead-Acid Storage Batteries, Leclanche-type Dry Cells and Batteries for Flash Lamps.

In preparing the draft standard, the ISI have given due weight to the existing foreign standards on the subject. Particular care has been taken to adapt these for conditions prevailing in India. While the specification relates essentially to cells and batteries for use in flash lamps, it can also be applied to the use of cells for certain general requirements. The standard contains definitions of terms used in industry, designations of cells and batteries, standard dimensions, requirements of workmanship, materials of construction, performance requirements, marking, rating and tests for initial voltage, initial and delayed life of cells.

Comments on the draft which has been widely circulated will be received up to December 31, 1949.

*Paints & Enamels* — The chemical composition and physical properties of different types of paints and enamels have been prescribed and special methods for testing their characteristics otherwise not included in the general specifications are detailed in the 69 drafts on ready-mixed paints and enamels.

Comments on these draft standards will be received by the Director till January 1, 1950.

*Oil Paints* — The 16 draft standards deal with chemical composition and physical properties of oil pastes for paints, including special methods for testing their quality, otherwise not included in the general specifications. Comments will be received up to January 5, 1950.

*Moisture Content of Timber* — "Draft Indian Standard Recommendations for Maximum Permissible Moisture Content of Timber for Different Purposes in Climate Zones" have been issued by the Timber Products Sectional Committee.



The object of this standard is to fix a maximum limit of moisture content for various stores to be used in the 4 climate zones into which the country has been broadly divided. The standard describes the areas covered by the 4 timber zones and the limits of maximum moisture content for 25 major varieties of timber stores. A map of India outlining the different climatic zones used for fixing the maximum moisture content for seasoned wood is also appended.

Comments on this standard will be received by the Director till January 15, 1950.

**Classification & Zonal Distribution of Timber** — For the purpose of this standard, India has been divided into 5 zones, keeping in view the political divisions of the country, the principal timber-consuming centres and the forest areas which feed them. The draft details the zonal distribution of all the common commercial timbers in India, classified according to their suitability for various categories of uses. In addition to trade names, botanical names and local names of commercial timbers in the several zones are given. A convenient map outlining the 5 zones is included.

Comments will be received by the Director till January 15, 1950.

### Bose Research Institute

THE ANNUAL REPORT (1948-49) of the *Bose Research Institute* reviews the research activities of the Institute under the following heads :

**Physics** — Valuable observations on cosmic rays have been recorded in the 4.5 litre high-pressure (11 atm.) steel ionization chamber of the Institute. High initial cosmic ray activity was observed on the 25th January 1949 during the period of intense sun-spot activity. Among the important additions to the equipment in this section are: (1) a small pressure ionization chamber for recording small atmospheric bursts; (2) Wilson chamber fitted with anti-coincidence circuit to enable tracks of light particles emitted from slowed-down mesons to be photographed; (3) photographic emulsion for plates recording tracks of ionizing particles as varied as  $\alpha$ -particles to fast electrons. In the field of nuclear physics, further progress is reported in the chemical separation of radioactive elements. A low-pressure ionization chamber has been constructed for studying nuclear

fission. Important ultrasonic investigations are being undertaken with the newly designed permanently evacuated glass triode valve MT 16 which generates electromagnetic oscillations of frequencies of the order of 1 megacycle per sec. A new apparatus for generation of atom ions is being tried since last year as the old generator constructed at the Institute was found unsuitable.

**Chemistry** — A method for the preparation of soluble salts of *Chincona febrifuge* in a dry amorphous state has been worked out. Synthesis of sesquiterpenes —  $\alpha$  and  $\beta$  selenines — has been effected. Interesting developments in the synthesis of *Penicillin amine* are reported. On the applied chemistry side, promising results have been obtained in the desulphurization of coal; a removal of 89.3 per cent of the total sulphur content in Ledo coal (Assam) has been effected. About 25 per cent of sulphur has been recovered in elementary form. *Impregnation of bleached jute yarn* with synthetic resins (of which *melamine resin* has been found most suitable) to restore the wet strength which it loses during bleaching, has been satisfactorily attained.

**Plant Physiology, Plant Breeding & Cytogenetics** — Work in this field includes recovery of the active (irritability) substance from *Mimosa pudica*, mechanism of transmission of excitation in *Mimosa pudica*, photo-synthesis in *Hydrilla*, relation between electric and mechanical responses in plants under tropic stimulation, nastic movements induced in plants by application of chemical growth substances, and nutritional experiments on cinchona plants. Cotton-breeding investigations for evolving long-stapled disease-resistant types suitable for cultivation in the delta areas of Bengal resulted in an increased yield of the hybrids in the F<sub>6</sub> generations. Manurial experiments showed that compost and cow-dung at the rate of 10 tons per acre gave best yields on *Aman* paddy representing an increase of 17 per cent over the control. X-ray irradiation and chemical treatment of jute being carried on under the scheme financed by the *Indian Central Jute Committee* has produced new mutant strains with desirable economic characters. Saline water experiments indicated that types in *C. Capsularis* could stand saline conditions.

Proposals for enlarging the scope of research activities of the Institute are among the main recommendations made by the *Vallarta Reviewing Committee*. For the implementation of these recommendations, the committee considered that capital grants of Rs. 1,70,000/- for building, Rs. 3,00,000/- for workshop and laboratory equipment and a recurring grant of Rs. 2,70,000/- per annum would be necessary.

The recurring grant received by the Institute during the year amounted to Rs. 1,00,000/- inclusive of allowance of Rs. 12,600/- and falls far too short of the amount required for effecting the proposed improvements in staff salaries.

Two grant-in-aid schemes, "On the application of synthetic hormones as weed killers" and "On the role of earthworms in relation to soil fertility" have been, on invitation, submitted to the ICAR by the Institute. Another scheme of investigation on oil seeds submitted last year to the *All-India Oil Seeds Committee* has been accepted and a grant has been sanctioned. A grant of Rs. 6,000/- has been received from *Burmah Oil Company* for palaeobotanical investigations. Of the old grant-in-aid schemes, those on "Physiological investigations on cinchona", and pilot-plant investigations on the economics of recovering quinine from the woody protein of the cinchona trees have been terminated, as the Health Ministry intimated their inability, in view of the present financial stringency, to renew the grants for the coming year. This work has had to be discontinued at a time when interesting results were being obtained.

The 10th Sir J. C. Bose Memorial Lecture was delivered by Prof. K. N. Bahl of the University of Lucknow on *Recent Advances in Our Knowledge of Nerves*.

### Indian Lac Cess Committee

THE ACTIVITIES OF THE INDIAN Lac Cess Committee have been reviewed in a recent booklet published by the Director, Indian Lac Research Institute.

The activities fall under two main heads: (1) organization of lac research, including the management and maintenance of the Indian Lac Research Institute, and the dissemination of the results of research with a view to their adoption by appropriate interests; and (2) improvement of the marketing conditions.

**Marketing of Lac** — With regard to the improvement of marketing conditions, the first act of the Committee was the institution of a Crop Statistical Section under a crop statistician. The second step in this direction has been the institution of marketing survey.

Recently, the Committee deputed the Director of the Indian Lac Research Institute to study the present position and future possibilities of lac abroad. The following significant points have come to light in the course of his visit to the U.S.A.: (1) synthetics have replaced lac in many of its known uses in the U.K. and the U.S.A. on price considerations alone; (2) stabilization of prices at a lower level is essential for maintenance of high level of exports; (3) for stabilizing prices and to meet post-war demands, cultivation of lac has to be extended; (4) funds should be provided for trade publicity in the U.S.A. and other consuming countries; (5) if India fails to meet overseas demand for lac at economic prices, Siamese lac industry, which is still in its infancy, is likely to undermine the position of Indian lac in foreign markets; and (6) only standard grades of lac are to be exported.

**Research** — Under the auspices of the Committee, researches have been conducted side by side on the life-history and bionomics of lac insects, and on the fundamental physico-chemical properties of lac, such theoretical studies being followed wherever possible by ancillary practical developments. On the entomological side, the work on the collection of broodlac has led to the development of a simple, accurate visual method of forecasting the swarming of lac larvae, thus enabling the cultivator to know the proper time to harvest the crop.

An important aspect of lac cultivation is the preservation of broodlac from the *Baisakhi* crop (maturing in June) and find means for protecting growing lac from the intense summer heat. Partial pruning of *ber* (*Zizyphus jujuba*), the host plant, before or after the infection of the *Baisakhi* crop and the partial defoliation of *palas* (*Butea frondosa*), another host plant, before infection, affords the necessary protection to the insects from the intense heat and prevents their untimely death. As much as 30 per cent saving in broodlac has been effected by this practice.

The above problem has been also solved by growing lac on ordinary non-lac hosts such as *Albizia lucida*, *Ficus bengalensis*, and *Ougenia dalbergioides*. The results achieved by this method have been quite encouraging.

The preservation of broodlac from the Jan.-Feb. crop on *kusum* (*Schleichera trijuga*) is different in that the larvae emerge late because of the cold and humid weather. This is overcome by effecting artificial emergence of the larvae in warm rooms and then transferring the brood on to the trees.

Another important finding in regard to the *kusmi* crop relates to the successful infection of *khair* (*Acacia catechu*) by the *kusmi* strain for growing *kusmi* lac. This alteration of *kusmi* brood with *khair* and vice versa results in increased production of the *kusmi* strain, which is known to produce superior quality of lac.

Other results of practical importance relate to the elucidation of the comparative role of the male and female insects in lac secretion and the asexual production of the females, the correct pruning methods and seasons of lac hosts, the use of suitable types of pruning instruments such as have been designed by the Institute, and cultivation of lac by the coupé system (this system ensures adequate periodic rest to the trees and thus adds not only to their life and vigour but also to their output). Systematic researches have also revealed the suitability of particular species of hosts for particular crops, the bad effects of self-infection of host trees, etc.

It is estimated that 40 to 50 per cent of lac crop is destroyed by enemy insects, which function as parasites or predators to the lac insects. The most important enemy is *Eublema amabilis* which accounts for more than half the total damage.

The following methods have proved efficacious in dealing with the predators: (1) encasing of broodlac sticks in 60-mesh wire-gauge baskets, which allow emergence of lac larvae but not of the enemy insects; (2) immersion of lac sticks in water for a prescribed time, which suffocates the predators to death; (3) collection of lac from lac sticks immediately after harvesting and killing the enemy larvae either by throwing them into fire or crushing; (4) not cultivating early-maturing and late-maturing varieties of lac simultaneously in the same locality, as

the enemy insects from the early varieties attack the late varieties and thus establish themselves better because of the continuous food supply.

**Fundamental Research** — Work has been done on such fundamental topics as the nature of the constituents of shellac, their molecular weight, basicity, etc. But the most outstanding results achieved in this latter field have been those relating to the nature of shellac in solutions. It has been established by studies on the viscosity, osmotic pressure, dialysis, etc., that, contrary to the previous idea which was based on its apparent behaviour, shellac is essentially non-colloidal, undergoing molecular dispersion at low concentrations. Its seemingly colloidal behaviour in relatively concentrated solutions has been traced back to its tendency to form clusters of solvated complexes with comparatively large surfaces which, serving as they do as seats of secondary forces, stimulate colloidal properties such as tendency to gel formation, high viscosity, etc. The idea of solvation has proved of great practical value in the discovery of "mixed solvents" for shellac. The discovery and the understanding of this phenomenon of "mixed solvents", besides resolving some previously known anomalies in shellac chemistry (e.g. precipitation on dilution), have been found to be of general validity and usefulness in the chemistry of other resins also.

The dielectric studies on lac and its constituent resins (hard and soft lac resins) have clarified the role of moisture in shellac and revealed its "non-tacking" property; incidentally it is this property that marks out its innate superiority over many synthetic resins in electrical applications.

**Applied Research** — A recent development of importance to be especially noted under this category is successful working out of the "autoclave method" for the manufacture of shellac. This method eliminates the use of cloth and charcoal, two costly materials, and does not require a solvent like alcohol; it is also less time-consuming, which all combine to make it much cheaper than the well-known indigenous process. Judged by the results of pilot-plant trials, its another advantage is that it is equally adaptable to both small-scale and large-scale operations.

In industry the scope of the applicability of lac has been definitely widened by entirely new developments in lac varnishes, plastics and adhesives. In the field of varnishes, for example, in spite of lac being an ideal ingredient of spirit-soluble varnishes, it had but restricted use in other types of varnishes, chiefly on account of its natural incompatibility with linseed oil. The problem has been solved in quite a number of ways: (1) use of linseed oil fatty acid instead of linseed oil itself, and reducing the acidity of the resulting product by treatment with glycerine; (2) esterification of linseed oil fatty acid itself with glycerine and combining the resulting mixed glycerides with lac; (3) cooking of certain proportions of metallic oxides, e.g. lime, red lead, etc., into linseed oil. These lac-oil combinations have been found eminently suitable as multipurpose varnishes of both baking and air-drying types which can be used as dressings for "oil-cloth" or "rexine", varnished cambric, insulating cloth (Empire cloth), home-recording discs, etc. Apart from these developments of lac-oil varnishes, substantial improvement has been effected in spirit varnishes, particularly with regard to their water resistance, elasticity, adhesion, etc., by (i) pre-treatment of lac with small amounts of chemicals like urea, maleic anhydride, etc.; (ii) condensation of lac with urea and formaldehyde; (iii) incorporation of small amounts of spirit-soluble nitro-cellulose; and (iv) condensing lac with cashew-nut shell liquid.

Other developments in lac varnishes have resulted from esterification of lac or aleuritic acid with monohydric alcohols followed by the action of polybasic acids, e.g. maleic or phthalic anhydride and fatty acids (e.g. linseed oil fatty acid). These new varnishes are found useful as coatings for electrical apparatus and resistances.

A spray gun for spraying hot molten shellac has been developed. The gun has been found excellent for coating wood, paper, metal, concrete, glass, asbestos, stoneware and various other surfaces. It is also found particularly useful for making laminated boards, tubes and such other electro-technical goods.

Another important use of lac is in the field of adhesives. A number of lac-based adhesives have been formulated. Aleuritic acid,

treated with phthalic anhydride, urea, formalin, etc., gives a transparent adhesive useful for making safety glass, laminated fabrics, etc. Shellac-formaldehyde-urea resin, originally developed as a moulding composition, has proved to be a good binder for the production of water-resistant plywood. Other compositions have been developed useful for making waterproof abrasive paper and grinding wheels which are already in commercial production. Flexible micanite, laminated hessian and paper, etc., are some of the other industrial products which can be made out of lac adhesives.

Other developments of interest to the industry include preparation of lac esters by reacting lac with mono- or poly-hydric alcohols or inorganic and organic acids including long-chain fatty acids. The resulting compounds may be used as plasticizers in polishes, sizes, waterproofing compositions, stoving varnishes, etc. Of these, lac-glycol ether esters, produced by reacting lac with ethylene glycol followed by esterification with poly-basic acids, give a series of products finding applications in electrical and other industries as adhesives, varnishes, rubber substitutes, etc. Esterification of lac-glycol ethers with rosin gives rise to glyptal substitutes. The use of sodium carbonate in place of borax in the preparation of fur-felt hat stiffeners is a war-time development deserving special mention.

A new method for the determination of moisture in lac has been developed. This method, based on the principle of simultaneous infrared heating and desiccation of a sample, reduces the time of moisture determination from the usual period of over 24 hr. to just 1 hr.

*Future of Lac* — At the moment Indian lac is facing a two-sided attack which, unless stemmed rightaway now, threatens to develop into a serious crisis. On one side, Siam, which was so long entirely dependent on India for her exports, has succeeded in producing high-grade shellac and her direct export to U.S.A. and other countries are regularly on the increase. On the other side, the threat from synthetic resins has now materialized, and lac has been completely ousted from the gramophone record industry in the U.S.A. To arrest further deterioration in the position of lac, three steps have been considered essential by the Lac Cess Committee: First, reorganization of the internal marketing system on

a rational basis, for which a move is already afoot; second: propaganda and publicity in the consuming countries. A Lac Sales Officer has been appointed in the United Kingdom with a view to establishing first-hand contact with the consumers of lac and ascertaining their difficulties such as may be due to uneconomic prices, inadequate or adulterated supplies. Liaison is also being maintained with the U.S. Shellac Importers' Association, which is expected to be further intensified in the shape of a joint publicity campaign to popularize lac in that country. The third move, perhaps the most important one judged by its long-term utility, is the reorganization of lac research along certain new lines. To develop the full potentialities of lac, fundamental research relating to its constitution is necessary. A beginning has already been made and the work on the constitution of lac now features as a major item of research under the Lac Cess Committee.

### Indian Standards Institution

THE SECOND ANNUAL REPORT OF the *Indian Standards Institution* for the year 1948-49 records considerable progress in its organizational and developmental activities. A new division, the Chemical Division Council, was inaugurated during the year. The number of committees and subcommittees functioning under the three existing Division Councils rose to 200. As many as 587 items approved for standardization are being investigated; at the close of the period under review, 209 draft standards are in advanced stages of development, while 9 have been published.

The ISI depends upon the specialized scientific agencies and institutions in the country, both governmental and private, for the preparation of the draft standards, it being not in a position to undertake such varied activities on its own. Some of these agencies are: Government Test House, Alipore; Geological Survey of India; Technological Laboratories, Matunga, Bombay; Indian Lac Research Institute, Namkum, Ranchi; Tata Research Laboratories, Jamshedpur; Delhi Cloth & General Mills Co. Ltd., Delhi.

The ISI committees responsible for the Secretariat of the *International Standards Organization (ISO)* for Shellac has finalized India's draft proposals on seedlac, shellac and bleached lac. The

ISO Technical Committee on Mica had finalized 2 draft proposals on grading and classification of mica which have now been circulated as draft Indian standards. These draft Indian standards will, after finalization, be submitted as Secretariat proposals for international standardization. Through efforts of the Bell Telephone Laboratories, the *ASTM* has, in recent years, developed objective methods of testing and classifying mica for electrical use. One of the features of these methods is that certain types of mica which, under the present visual classification system, come under lower grades, could be upgraded. One of the tasks recommended for the *ISI* committee on the subject is to examine these methods for use by the Indian producers.

The Bell Telephone Laboratories have prepared a film demonstrating the operation of these methods and a copy of this film together with a set of mica samples demonstrating the upgrading process has been loaned to the *ISI* for 2 years.

During his recent visit to the U.K., the U.S.A., Canada, Switzerland and other Continental countries, the Director of the *ISI* discussed with the Indian Trade Commissioners in Canada and U.S.A. the question of standardization of products exported from India to these countries. In addition to shellac and mica, the major items of Indian export were : jute, carpet wool, coir products, carpets, manganese ore, chromium ore, sports goods.

In the opinion of the Indian Trade Commissioners abroad, the absence of standards has been particularly felt in relation to carpet wool and floor coverings including coir matting. The Commerce Ministry was contacted to determine the extent of interest, to obtain technical data and to compile a list of parties concerned with the manufacture and export of these goods. The *ISI* has, in this context, decided to set up a special Committee for Control of Quality of Export Goods to formulate specific recommendations for the inspection and testing of important items of export and legislation required to enforce such a control.

Under the scheme for certification marking, which is now under consideration, the *ISI* feels that the following commodities require immediate attention with regard to their standardization for export

market : tobacco, sunn hemp, skins (goat and sheep), bristles, kapok, hair wool, oil seeds, vegetable oils, seedlac, shellac, coir and coir products, tea-chests, cotton textiles (a) seaworthy packing for cotton textiles, mica, manganese ore and sports goods.

In addition to these commodities, the question of standardizing all kinds of floor coverings produced in India has recently been raised. Indian carpets used to find their way into export trade to a large extent in the past, but it is understood that this trade is fast dwindling, chiefly due to the fall in quality of goods more recently exported. In addition to quality consideration, there is the important question of developing new designs and styles to suit the taste of the foreign consumers. Little or no attempt has been made in an organized manner by the Indian industry to determine the tastes and needs of their foreign customers. In order to revive this trade, it is not only necessary to consider standardization, but also the organization of an agency to investigate the problems of design and styling and to advise the Indian producers.

It has been proposed that the organization of a large export corporation mainly financed and controlled by the Government with the object of : (1) co-ordinating production and procurement of floor coverings from all parts of India in collaboration with provincial and state Directors of Industries ; (2) investigating tastes of foreign consumers to guide designs and styling ; (3) organizing training of skilled workers ; (4) assisting the *ISI* in developing standards for cottage-industry products of this nature ; (5) supplying technical guidance, data on preferred designs, raw materials, and tools to cottage-scale producers ; etc.

The Institution operated well within its allotted budget during the period under review. Although the year's balance exceeds the anticipated opening balance of the next year by Rs. 50,000, the Institution is likely to face a budget deficit of Rs. 1,44,675 during 1949-50 due to a rapid expansion in its activities. In view of this, the Government have been requested to increase the grant-in-aid from Rs. 2 lakhs to Rs. 4.5 lakhs for the next financial year. Similarly, the Commerce Ministry have been approached for an annual grant of Rs. 25,000 to implement the decisions regard-

ing the standardization of important items of export.

### Announcements

*Indian Science Congress, 1950 Session* — The 37th session of the Indian Science Congress Association is due to be held at Poona from January 3 to 8, 1950, under the presidency of Professor P. C. Mahalanobis. A number of distinguished foreign scientists from the U.K., the U.S.A., France and the U.S.S.R. have been invited. Prof. Desmond Bernal of Birkbeck College, University of London, Sir Robert Robinson, President of the Royal Society, accompanied by Lady Robinson, Prof. Joliot-Curie and Madame Joliot Curie, Prof. Herman Mark, who is an authority on polymer chemistry, and Dr. E. U. Condon, Director of the National Bureau of Standards, Washington, are expected to attend the session. It is also hoped that Prof. W. A. Engelhardt, of the Pavlov Institute of Physiology, Academy of Sciences, U.S.S.R., will attend the session.

*Nobel Prize for Awards (1949)* : *Chemistry* — Prof. F. W. Giauque, Berkeley, California ; *Physics* — Prof. Hideki Yukawa, Japan ; *Physiology and Medicine* — shared by Dr. Rudolf Hess, Zurich University Institute of Physiology, and Prof. Maritus Antonio Egas Moniz, Lisbon University.

*Fritzsh Award* — Sir John L. Simonsen, Director, *Colonial Products Research Council*, has been awarded the American Fritzsh Award by the American Chemical Society. The award is instituted to recognize and encourage outstanding achievement in analysis, research and new applications of essential oils, essential oil isolates and related chemicals. Sir John is the first recipient of the award.

*Bibliographical Series of the Science Library of the Science Museum, London*, have been received in the UNESCO Office, University Buildings, Delhi. Copies of the bibliography on specific subjects can be duplicated and supplied on request.

*Publications of International Scientific Union* — Few copies of (1) Repertory of Cosmic Rays laboratories and physicists and (2) Report on the Principles of Rheological Nomenclature, are available from UNESCO Office, University Buildings, Delhi.

(Continued on page 507)

# Reports from States & Provinces

## MYSORE

### Occupational Institutes

AN OCCUPATIONAL INSTITUTE to provide a diploma course in oil and soap technology and artisan courses in electric wiring, tailoring and spinning (textile) has been opened in Devangere.

The Government have also sanctioned the starting of similar occupational institutes in Mysore City, Chintamani (Kolar district), Bhadravati (Shimoga district) and Hassan.

The institute at Hassan will provide a diploma course in metal technology, artisan courses in electric wiring, auto mechanics, tailoring and dress-making, pottery and rattan work. The Chintamani institute will have, in addition, a diploma course in silk and woollen technology.

### Development of Mainad Areas

THE CENTRAL MINISTRY OF AGRICULTURE is taking concrete steps in co-ordination with the provincial governments concerned to develop the 21,000 sq. miles of territory comprising North Kanara district of Bombay, South Kanara district of Madras, and parts of Coorg and Mysore.

The immediate aspect of the programme of development is to improve food production in some of these parts by the use of manures, better seeds, etc., and bringing under cultivation fallow land by heavy tractors.

The second, the short-term aspect, is colonization which is to be achieved by removing the malaria menace which has been responsible for de-populating the area. Finally, the long-term aspect of the plan includes setting up of forest industries, opening up of communications, development of ports and rubber plantations in this region.

A Planning Committee consisting of experts from the Centre, the various provinces and the states concerned, is surveying the area on the above lines.

## TRAVANCORE & COCHIN

### Aluminium Industries, Kundara

THE FIRST FACTORY IN INDIA to manufacture aluminium cables, both plain and steel reinforced, and cast aluminium electrical accessories, is being set up at Kundara in Travancore.

The factory is equipped with latest wire drawing machines capable of drawing wire of thicknesses from  $\frac{1}{8}$  in. to  $\frac{3}{16}$  in. dia. at speeds ranging from 1,000 to 2,000 ft. per min. The drawn wires are loaded on to cabling machines — "stranders", which strand steel wires on aluminium wires or vice versa to make a composite cable. The larger cables, having a diameter up to  $\frac{1}{2}$  in., are capable of carrying hundreds of thousands of h.p. of electricity over long distances.

The aluminium foundry attached to the wire mill is fitted with an electrically heated forced-air circulating furnace of the vertical-pit type used for solution heat treatment and precipitation and ageing treatments of aluminium alloys. This will enable the production of high-strength alloy accessories.

A laboratory equipped with a Hooper conductivity bridge, a metallographic microscope and camera and other apparatus ensures inspection and controls the quality of the products at different stages.

The factory will manufacture 2,000 tons of finished cables annually and will substantially meet the country's requirements of the cables in the multi-purpose river and valley schemes which are now either in the planning stage or under execution.

## NEW DELHI

### Indian Oil Seeds Committee

MEASURES TO INCREASE THE PRODUCTION of oil seeds, improvement of marketing and distribution methods, and promotion of export trade in oil seeds were the principal items discussed during the

5th meeting of the Committee held in October.

The President drew the attention of the Committee to the dwindling exports of oil seeds. Only 2 lakh tons were exported in 1948-49 as against 8-10 lakh tons during the pre-war period. This was accounted for by high prices and higher internal demand. Increased production by careful crop planning and intensive cultivation was urged to bring down prices and stimulate exports especially to dollar areas with a view to take full advantage of devaluation of the rupee. The Committee accepted the principal recommendations of the Export Promotion Committee on the export of oil seeds.

The establishment of a village oil industry institute at Nagpur to provide training in oil industry and undertake research work on improved *ghanies*, was recommended for developing the village oil industry. Co-operative societies of village oil crushers in various provinces and states were also suggested. A scheme of multiplication and distribution of improved strains of groundnut in Bombay and various research schemes on oil seeds in other provinces and states were recommended. A number of schemes for prevention of pests and diseases were also considered.

### New Steel Plants

RECOMMENDATIONS ON THE LOCATION and working of new steel plants have been forwarded to the Government by the three foreign consultants appointed for the purpose.

Keeping in view adequate transport, water supply, decentralization and strategic importance, the consultants have unanimously recommended location of one of the plants in C.P. and the other in Orissa, each with a capacity of half a million tons. Two plants instead of one are favoured because of flexibility in production, rapid construction leading to early production, and facility for doubling at a later date. The capital costs for each plant have been estimated between Rs. 50 to 60 crores. Each plant is expected to take 4 to 5 years for completion.

According to the report, India's 1953-55 demands for iron and steel which will be of the order of 2.7 to 3.4 million tons annually, thus leaving a deficit of not less than a million tons (existing productions capacity being 1.3 million

tons), confirms the necessity for new steel plants with a capacity not less than 1 million tons. This takes into account the expansion plans of existing producers.

India's reserves of iron ore, coalfields at Jharia (which are supposed to give the best coking coal), limestone, dolomite, manganese and fluorspar are adequate for working the plants provided stringent conservation and utilization of these researches is ensured.

## MADRAS

### The Oil Technological Institute

THE FOUNDATION-STONE OF "THE Oil Technological Institute", the first of its kind in the province, was laid by the Minister for Revenue and Labour at Anantapur on August 26.

The Institute, estimated to cost initially Rs. 6,29,850, has been designed on modern lines and will have research and special pilot laboratories equipped with up-to-date appliances. Its first session is expected to commence in 1950.

The object of the Institute is to train apprentices leading to a diploma course in oil technology; to undertake research work on solvent extraction of oils; fat splitting and manufacture of fatty acids and their metallic compounds; manufacture of groundnut butter; essential oils; plastics powder and moulding; and cracking of vegetable oils for motor spirit. It will also deal with other problems relating to the production and processing of oils and oil industries.

Having regard to the importance of oil seeds, especially of groundnuts (of which there are 4 million acres under cultivation) in the economy of the province, the establishment of the Oil Technological Institute should provide bright chances of a speedy development of this area.

## UNITED PROVINCES

### Development of Cottage Industries

THE U.P. COTTAGE INDUSTRIES Department organized a conference of technicians and technologists in celebration of the cottage industries week in September.

The conference adopted reports of 8 of its subcommittees dealing with 15 different industries including handloom, dyeing and printing, carpet weaving, wool grading, fibres, hand-made paper, essential oils, cottage oil, ceramics, non-ferrous metals, tinning, electroplating palm and cane and lac.

Recommendations of these committees deal with research in, and investigation of, technical problems; commercial exploitation of problems already satisfactorily solved in the laboratory and standardization of the products of various industries to be undertaken by the Indian Standards Institution and other Government agencies. A number of recommendations have been aimed at the improvement of the quality of the goods and exploration of fields of new industrial endeavour to stimulate the export trade of the country. For instance, it has been suggested that the Indian carpets be so standardized as to attract the foreign buyer, the quality of sunn hemp be improved for export and for furthering its utility in India; the essential oil industry may concentrate its efforts on increased production of standardized products for which India is in a specially advantageous position.

The conference has urged expeditious commercial exploitation of linseed fibre which can be converted into a useful substitute for jute but still continues to be consumed as a fuel.

## NOTES & NEWS

(Continued from page 505)

*American Electroplaters' Society (A.E.S.)* has offered its Research Report published to date at a group price of \$ 5.00. This represents a saving of \$ 4.05 for a total of 14 reports which include the following: Adhesion of Electrodeposits, Determination of Impurities in Electroplating Solutions, Stripping of Copper from Various Base Metals, Porosity of Electrodeposited Metals, Disposal of Plating Room Waste, Method of Testing Thickness of Electrodeposits, Physical Properties of Electrodeposits, and Effect of Impurities and Purification of Electroplating Solutions. The re-

ports are available from the Society's Office at 473 York Road, Jenkintown, Pa.

*International Electrodeposition Conference* — The 37th Annual Convention of the *American Electroplaters' Society* which is to be held from June 11 to 15, 1950, in Boston, Mass., U.S.A., will also be the venue for the 4th *International Electrodeposition Conference* which is being held with the collaboration of the *Electrodepositors' Technical Society* of England.

*American Electrochemical Society* — At a symposium on "Electroorganic Chemistry", held in Chicago during October, Dr. B. B. Dey presented a paper on "Electrolytic Reduction of Aromatic Nitro Compounds in Acid and Alkaline Media". The work reported in the paper is the outcome of a scheme of research financed by the *Council of Scientific & Industrial Research*.

*British Association for the Advancement of Science* — The annual meeting of the British Association in 1951 will be held in Edinburgh under the presidency of H.R.H. the Duke of Edinburgh. Sir Harold Hartley has been elected president for the year 1950.

*Mathematical Society* — The 16th Conference of the Indian Mathematical Society will be held in Madras under the auspices of the University of Madras on 26th, 27th and 28th December 1949.

*Commonwealth Research Grants for India* — The British Council has, in pursuance of the scheme established last year at the Congress of Universities of the Commonwealth, awarded travel grants to two Indians for studies at British universities. The beneficiaries are: Mr. O. P. Bhatnagar of Allahabad University and Mr. B. Bhattacharya of Hindu University, Banaras.

*Indian Central Cotton Committee* — The following have been appointed as members of the Indian Central Cotton Committee: Shri R. G. Saraiya; Shri Madanmohan Mangaldas; Shri G. M. Kothari; Shri A. K. D. Balarama Raja; Shri Narayan Dass Mukerjee; and Seth Bhogilal M. Shah.

# INDIAN PATENTS

The following is a list of a few of the Patent Applications notified as accepted in the *Gazette of India*, Part II, Section 1, for October 1949.

## Plastics & Plasticisers

39982. ELECTRICAL & MUSICAL INDUSTRIES LTD. : Gramophone records : *Comprising shellac as thermoplastic ingredient and a bituminous substance in admixture therewith whereby separation is prevented.*

## Inorganic Chemicals

40889. ANGLO-IRANIAN OIL CO. LTD. : Catalysts : *Reacting cobalt nitrate and ammonium molybdate in excess of ammonia and mixing the solution with anhydrous porous gamma alumina.*

## Organic Chemicals

39648. I.C.I. LTD. : Terephthalic acid : *Passing oxygen into liquid p-xylene containing an oxidation catalyst.*

## Miscellaneous Chemicals

39616. I.C.I. LTD. : Process for the manufacture of leuco sulphuric ester derivatives of vat dyestuffs : *Sulphation is carried out in the presence of an organic amide in which hydrogen atoms attached to the amide nitrogen atom have been replaced by hydrocarbon radicals.*
39620. I.C.I. LTD. : New dyestuffs : *Sulphating in the presence of an organic amide in which the hydrogen atoms are attached to the amide nitrogen atom.*
39741. I.C.I. LTD. : New hydroxy fatty acid ester ethers : *Converting estrolides of hydroxy fatty acids into their ester-ethers with ethylene glycol and or polyethylene glycol.*

## Drugs & Pharmaceuticals

39282. ELI LILLY & Co. : Improvements in or relating to process of obtaining crystalline salts of penicillin with sodium, potassium or ammonium : *Transforming the aqueous solution of such salt that is obtained directly from the organic solvent extract of a penicillin broth into a*

*reduced volume solution of such salt in a solvent of which 95 per cent is an organic solvent.*

## Metals & Metal Products

39893. MEEHANITE METAL CORP. : White cast iron castings : *Preparing molten cast iron which produces a constitutional carbide value of  $1\frac{1}{2}$  to  $7\frac{1}{2}$  times white and processing it to produce a processed carbide value of 1 to  $2\frac{1}{2}$  times white.*
40271. G.E.C. LTD. : Cold welding of ductile metals : *Cold pressure welding of two ductile metals of different hardness wherein area of applied pressure is greater for the metal of lower hardness.*

## Printing, Publishing & Allied Industries

39615. I.C.I. LTD. : Preservation of rubber latex : *Mixing latex with a colloid stabilizer and a neutral bactericide.*

## Textile & Textile Products

39617. I.C.I. LTD. : A new white fluorescent substance for textile treatment : *Reacting cyanuric chloride with diaminostilbene disulphonic acid and diethanolamine.*
39618. *Reacting cyanuric chloride with diaminostilbene disulphonic acid and monoethanolamine.*
39619. *Reacting cyanuric chloride, diaminostilbene disulphonic acid, monoethanolamine and an amine.*
39886. THE DAYTON RUBBER COMPANY : A method and device for impregnating textile materials with liquid : *Consisting of a pair of co-operating rollers one of which immerses into a sizing bath and the other is provided on the outside with sponge-like structure.*
39793. I.C.I. LTD. : New dyestuffs for dyeing wool by the chromate process : *Comprises coupling diazotized 5 sulphoanthranilic acid with 1-(p-2 ft. or 4 ft. chlorophenoxyphenyl) -3-methyl-5 pyrazolone.*

## Exhibition of Patents & Inventions and Designs & Trade Marks

THE LORD REAY MAHARASHTRA INDUSTRIAL Museum, Poona, is organizing an Exhibition of Patents & Inventions and Designs & Trade Marks in January 1950, on the occasion of the Session of the Indian Science Congress which is going to be held in Poona.

The last exhibition on this subject was organized by the Museum three years ago. The Exhibition opens on the 1st of January 1950 and concludes on the 3rd of March. The charge for exhibiting will be Rs. 20 per exhibit. The exhibits will be received up to 20th December 1949.



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# AROMATIC PLANTS OF INDIA

## PART IX

BY

S. KRISHNA & R. L. BADHWAR

FOREST RESEARCH INSTITUTE, DEHRA DUN

FAMILY

40. Leguminosae



Journal of Scientific and Industrial Research, Delhi

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## **NATIONAL INSTITUTE OF SCIENCES RESEARCH FELLOWSHIPS**

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# Aromatic Plants of India

## Family XL — LEGUMINOSAE

(Pulse or Bean Family)

THIS is one of the most cosmopolitan and the second largest family of flowering plants, with about 500 genera and 12,000 species of herbs, shrubs, lianas, and trees. The flowers are usually irregular and papilionaceous (like that of pea) or rarely regular. The fruit is a true pod or legume.

The family comprises three subfamilies; the largest is the Papilionatae which is cosmopolitan and the other two, Caesalpinioideae and Mimosoideae, are found mostly in the tropical and warm temperate zones. These subfamilies are nowadays recognized by botanists to constitute distinct families, but for the sake of convenience are being dealt with together under Leguminosae in this work.

The Leguminosae, taken as a whole, is the most important family in India from the economic point of view. A large number of species yield food, fodder, drugs, insecticides, fibres, gums and resins, fatty oils, tanning materials, dyes, timbers, etc., and many others are grown in gardens and avenues as ornamental plants, shade trees and for hedges. A very interesting feature, which gives to this family an outstanding importance, is the power of fixing the free nitrogen of the air by means of the peculiar bacterial organisms, present in the root-nodules of many plants, thus automatically rejuvenating the soil on which they grow. This fact has been taken advantage of in economic agriculture.

The seeds of many plants of the family are edible, and some of the familiar ones are: groundnut or peanut (*Arachis hypogaea* Linn.), gram or chick-pea (*Cicer arietinum* Linn.), pea (*Pisum sativum* Linn.), pigeon pea or *harhar* [*Cajanus cajan* (Linn.) Millsp., syn. *C. indicus* Spreng.], lentil or *masur* (*Lens esculenta* Moench, syn. *Ervum lens* Linn.), urd (*Phaseolus mungo* Linn., var. *roxburghii* Prain), *mung* (*Phaseolus radiatus* Linn.), French bean or kidney bean (*Phaseolus vulgaris* Linn.), Lima bean or Duffin bean (*Phaseolus lunatus* Linn.), *moth* (*Phaseolus aconitifolius* Jacq.), *rawan* or *barbatti* [*Vigna sinensis* (Linn.) Savi ex Hassk.,

syn. *V. catjang* Walp. and *V. catiung* Fl. Brit. Ind.], soya bean [*Glycine max* (Linn.) Merr., syn. *G. soja* Sieb. & Zucc.], broad bean (*Vicia faba* Linn.), chickling vetch or *khesari* (*Lathyrus sativus* Linn.), etc. The young pods of *sem* (*Dolichos lablab* Linn.), jack bean or horse bean [*Canavalia ensiformis* (Linn.) DC.], cluster bean or *guar* [*Cyamopsis tetragonoloba* (Linn.) Taub., syn. *C. psoraloides* DC.] and some species of *Phaseolus*, *Vigna*, etc., are also extensively eaten in this country. The leaves of fenugreek or *methi* (*Trigonella foenum-graecum* Linn.) are commonly eaten as a vegetable. Horse gram or *kooltee* (*Dolichos biflorus* Linn.), lucerne or alfalfa (*Medicago sativa* Linn.), clovers and sweet clovers (species of *Trifolium* and *Melilotus*) are some of the famous fodder plants. Kudzu bean [*Pueraria thunbergiana* (Sieb. & Zucc.) Benth.] of Japan and E. Asia has been recently introduced into India. It is a perennial vine which yields good fodder. A valuable starch is prepared and marketed in E. Asia from its large tuberous roots, which, in favourable climates, are said to attain a weight of over 80 lbs.

As sources of fatty oils or solid fats, groundnut, soya bean, and *karanj* [*Pongamia pinnata* (Linn.) Merr., syn. *P. glabra* Vent.] are well known.

Important dyes are obtained from the indigo plant (*Indigofera tinctoria* Linn.), *palas* [*Butea monosperma* (Lam.) Kuntze, syn. *B. frondosa* Koen. ex Roxb.], sappanwood (*Caesalpinia sappan* Linn.), red sanderswood or red sandalwood (*Pterocarpus santalinus* Linn. f.), barwood or camwood (*Baphia nitida* Afzel ex Lodd.) of Africa, logwood (*Haematoxylon campechianum* Linn.), etc. Logwood is the source of the well-known laboratory stain haematoxylin, and is sometimes cultivated in Indian gardens.

The dried decoction of khairwood [*Acacia catechu* (Linn.) Willd.] is the *katha*, which is an important ingredient of *pan* or betel-leaf preparation. *Katha* consists of catechin which is used in medicine, and catechu which is used for dyeing, especially brown and composite shades on cotton, tanning,

preserving fishing nets, etc. Similar preparations are also made from *Acacia chundra* Willd.

Like the wattle bark from *Acacia mollissima* Willd., the barks of babul [*Acacia arabica* (Lam.) Willd.] and tanner's cassia, *avaram* or *tarwad* (*Cassia auriculata* Linn.) are among the most important tanning materials of India. The pods of divi-divi or American sumach [*Caesalpinia coriaria* (Jacq.) Willd.] are rich in tanning materials; this plant is often cultivated in India.

One of the most important commercial gums is obtained from a plant of this family. This is the gum tragacanth from *Astragalus gummifer* Labill. and other species of *Astragalus*. The gum arabic is obtained from *Acacia senegal* (Linn.) Willd. and the gum babul from *A. arabica* (Lam.) Willd. *Pterocarpus marsupium* Roxb. is the source of kino, an astringent gum used in medicine and also for dyeing and tanning purposes. The mesquite gum is obtained from the American *Prosopis glandulosa* Torr. and *P. chilensis* (Molina) Stuntz [*P. juliflora* (Sw.) DC.], which are cultivated in India. Tragacanth is derived from the pods of the Syrian carob tree (*Ceratonia siliqua* Linn.). Attempts have often been made to cultivate the carob tree in India, but have not on the whole succeeded very well. The tree fruits very scantily under Indian conditions.

Several African trees yield copals (hard resins), which are used in making varnishes. The Zanzibar copal and the closely allied Madagascar and Mozambique copals are obtained from *Trachylobium verrucosum* (Gaertn.) Oliver. Zanzibar copal is the hardest of all resins, except amber, and is very valuable. The resin exudes naturally from the trunk, branches and fruit, but most of the commercial supply is obtained from the semi-fossil material from the still living trees, and the fossil material from the trees which no longer exist. Congo copal is derived from *Copaifera demusei* Harms and *C. mopane* J. Kirk ex Benth. of the Congo basin. Sierra Leone copal is obtained from *C. guibourtiana* Benth. and *C. salikounda* Heckel of Sierra Leone. The S. American copal is derived from the S. American locust (*Hymenaea courbaril* Linn.); it is the softest of all copals, and consequently the least valuable.

The acid fruit-pulp of tamarind (*Tamarindus indica* Linn.) is extensively eaten in India, and the seed kernels are largely used for sizing purposes. The kernels are by

far the cheapest source of a "pectin", called jellose, which, though differing chemically from the fruit pectins, is better than the latter in performance in so far that it sets to a jelly with sugar even without acids.

Sunn hemp from *Crotalaria juncea* Linn. is a valuable fibre, and so is *jayanti* [*Sesbania bispinosa* (Jacq.) Fawcett & Rendle, syn. *S. aculeata* (Willd.) Pers.]. Solar hats or *sola topees* are made from the spongy stems of *sola* or pith plant (*Aeschynomene aspera* Linn.), a stout aquatic perennial plant.

Considerable trade is done in the saponaceous pods of *shikakai* [*Acacia concinna* (Willd.) DC.] which are used for shampooing.

Many plants, such as sissoo or *shisham* (*Dalbergia sissoo* Roxb.), Indian rosewood (*Dalbergia latifolia* Roxb.), ironwood [*Xylia xylocarpa* (Roxb.) Taub., syn. *X. dolabrifolia* Benth.], siris [*Albizia lebbek* (Linn.) Benth.], Andaman padauk (*Pterocarpus dalbergioides* Roxb., syn. *P. indicus* Baker, in part, non Willd.), Burma padauk (*Pterocarpus macrocarpus* Kurz), red sanderswood or red sandalwood (*Pterocarpus santalinus* Linn. f.), *bijasal* (*Pterocarpus marsupium* Roxb.), babul [*Acacia arabica* (Lam.) Willd.], American ebony, coccoloba wood or granadillo [*Brya ebenus* (Linn.) DC.], Australian blackwood (*Acacia melanoxylon* R. Br.), etc., yield valuable timber; the last-mentioned is extensively planted in the Nilgiris. Recently, during the World War II, when the supplies of *lignum vitae* for the manufacture of marine propeller shafts became scarce, the wood from the Indian *Acacia chundra* Willd. was considered to be an excellent substitute and a considerable demand for it has arisen.

Of the most familiar plants found in gardens and along roadsides in India may be mentioned, in addition to the several of the above, the sweet pea (*Lathyrus odoratus* Linn.) with various coloured flowers; *gul mohur* or gold-mohur tree [*Delonix regia* (Boj. ex Hook.) Rafin., syn. *Poinciana regia* Boj. ex Hook.] with red flowers; *asoka* tree (*Saraca indica* Linn.) with brilliant orange-scarlet, fragrant flowers; coral tree [*Erythrina variegata* Linn., var. *orientalis* (Linn.) Merr., syn. *E. indica* Lam.] with brilliant scarlet flowers; Indian laburnum (*Cassia fistula* Linn.), *sau* [*Albizia chinensis* (Osbeck) Merr., syn. *A. stipulata* Boiv.] grown for its shade and for protecting tea plantations; *colvillea* (*Colvillea racemosa* Boj.) with bright-orange flowers; peacock flower

or Barbados pride [*Caesalpinia pulcherrima* (Linn.) Sw.] with scarlet or yellow flowers; species of *Bauhinia*, etc.

The family contains the largest number of plants which are poisonous to fishes. Many of these, which are spread over 27 genera, are used as insecticides. The most important of these are some species of *Derris*, such as *D. elliptica* (Roxb.) Benth. (tuba), from the roots of which an active insecticidal principle, rotenone, has been isolated. Rotenone has also been isolated from members of the genera *Lonchocarpus*, *Millettia*, *Tephrosia*, and others. The aromatic fenugreek or *methi* (*Trigonella foenum-graecum* Linn.) is used as an insect repellent for stored grains in the Kangra district of the Punjab.

The family contains a large number of medicinal plants, but only some of the more important ones are mentioned below. Liquorice root (*Glycyrrhiza glabra* Linn.) is largely used as a remedy for cough, as a purgative, and also to mask the taste of bitter and nauseous medicines; it contains the glycoside glycyrrhizin. Balsam of Tolu is obtained from *Myroxylon balsamum* (Linn.) Harms (*M. toluiferum* H. B. & K.), a native of Venezuela, Columbia and Peru, and contains an essential oil; the balsam is much used as an expectorant, and as an ingredient in cough lozenges. The Calabar or ordeal bean (*Physostigma venenosum* Balf.), a tropical W. African climber, contains several alkaloids of which eserine (physostigmine) is the most important. The alkaloid is used in ophthalmic practice to contract the pupils. Senna leaves and pods from *Cassia angustifolia* Vahl and *C. acutifolia* Delile are well known as mild purgatives. The pulp of the pod of Indian laburnum or purging cassia (*Cassia fistula* Linn.) is used as a mild household laxative in India. *Acacia senegal* (Linn.) Willd. and *A. arabica* (Lam.) Willd. are the sources of gum arabic and gum babul which are commonly used for emulsifying oily preparations, but have no medicinal value. *Astragalus gummifer* Labill. and other species of *Astragalus* of W. Asia yield gum tragacanth, which is used as an excipient for pills and for emulsions to suspend heavy powders in water; none of the species yielding this gum is found in India. The gum kino from *Pterocarpus marsupium* Roxb. is used as an astringent, particularly for inflamed throats and as a mouthwash for spongy gums. The European broom (*Cytisus scoparius* Link), which has

been introduced in the Nilgiris (Ootacamund), is used in Western medicine, chiefly as a diuretic. It contains sparteine and other alkaloids, and a soluble phenol, scoparin.

Some plants are known to yield essential oils, while some others, though odoriferous, still remain uninvestigated.

The yellow, strong-scented flower-heads of *Acacia farnesiana* (Linn.) Willd. constitute the cassie flowers which yield the cassie perfume of commerce. This perfume is very delicate, and is usually extracted in the form of a pomade. The yellow, sweet-scented flower-heads of the Australian *Acacia dealbata* Link and of some other species of *Acacia* are used for the preparation of the well-known mimosa perfume. Both these species are described in detail later.

Sweet pea (*Lathyrus odoratus* Linn.) is a well-known garden plant having elegant, sweet-scented flowers. Natural perfume from these flowers is not extracted on any large scale, and the bulk of the sweet-pea perfumes of commerce is synthetic.

The flowers of the Spanish broom (*Spartium junceum* Linn.), a European shrub which is also sometimes cultivated for its fibre, have a powerful pleasant odour, which changes quickly after the flowers are cut. They are treated in Grasse by volatile solvents, and on rare occasions by maceration in a pomade. The extraction products of these flowers are among the precious raw materials for perfumery. The yield of petroleum ether concrete, which has a waxy aroma, varies between 0.09 and 0.18 per cent., and this concrete gives 30 to 40 per cent. of absolute. The concrete has congealing point 49.8° and saponification val. 44.8. The absolute has acid val. 33.6 and ester val. 85.9. A distillate having ester val. 101.1 has also been obtained in a yield of 5 per cent. (Naves & Mazuyer, 1947. *Natural Perfume Materials*: p. 170).

The flowers of another foreign broom (*Genista tinctoria* Linn.) have yielded 0.161 per cent. of a petroleum ether concrete, which gave 53 per cent. of absolute and contained 2.26 per cent. of yellowish steam-distillable products with heavy odour (Naves & Mazuyer, *loc. cit.*).

The S. American trees *Dipteryx odorata* (Aubl.) Willd. and *D. oppositifolia* (Aubl.) Willd. are the source of the well-known tonka beans, those from the latter (*Para* beans) being smaller and not so valuable

as the former (Angostura beans). The tonka beans contain about 3 per cent. of coumarin, which is of considerable importance in the manufacture of perfumes. The beans, and their extract are used for flavouring snuff, tobacco, and sachet powders, and as a substitute for vanilla in cocoa, candy, and ice cream. Although *Dipteryx odorata* is the most important natural source of coumarin, the latter is found in several species of *Dipteryx*, *Myroxylon*, *Melilotus*, etc., belonging to this family, and also a number of plants of other families. The tonka beans have, however, been largely replaced by synthetic coumarin. For a detailed account of tonka beans the reader is referred to an article by Albes (1916. *Amer. Perfum.*, 10 : 278).

The reddish-brown, thick, syrupy, viscous balsam of Peru, obtained from the Central American tree *Myroxylon pereirae* (Royle) Klotzsch, is of great value as a fixative in the preparation of heavy odours of Oriental type, and is a desirable addition to all perfumes intended for lipsticks owing to its soothing qualities. The balsam, which has a penetrating and very sweet odour, contains 55 to 65 per cent. of cinnamein (a mixture of benzyl benzoate and benzyl cinnamate), small amounts of free aromatic acids, benzyl alcohol, nerolidol, and a trace of vanillin. When pure it has sp. gr. 1.1400 to 1.6120 and  $n_D^{20}$  1.5800 to 1.5855. The balsam is now available as a water-white viscous oil for use in good-quality perfumes.

Apart from the balsam there is also an essential oil in the wood of *Myroxylon pereirae*. This oil, which has recently been reported by Naves (1948. *Perfum. essent. Oil Rec.*, 39 : 280), can be obtained by steam distillation of the wood of even those trees which have been tapped for the balsam. The oil is recovered in a yield of 0.736 to 1.08 per cent., and its main constituent is free nerolidol (50 to 70 per cent.), together with cadinenes and free cadinol. This finding points out a commercial source of nerolidol, which has a demand in high-class perfumery and which has been scarce so far. According to Naves (1947. *ibid.*, 38 : 191), nerolidol is also obtainable from the essential oil (cabreuva oil) from the wood of the Brazilian species *Myrocarpus frondosus* Allem. and *M. fastigiatus* Allem. to an extent of 60 to 80 per cent. Further, according to him, nerolidol can be converted into farnesol, another rare ingredient used in perfumery. ❀

The balsam of Tolu, a brown or yellowish-brown plastic substance with a sweet hyacinth-like fragrance, is obtained from *Myroxylon balsamum* (Linn.) Harms (*M. toluiferum* H.B. & K.) of Venezuela, Columbia, and Peru. Considerable amounts of it are used as a fixative in perfumery and for flavouring cough syrups. It contains up to 80 per cent. of resin, together with benzoic and cinnamic acids, vanillin, benzyl benzoate, benzyl cinnamate, and a small quantity of volatile oil. A genuine balsam of Tolu has an acid value from 92 to 135 and an ester value of 59 to 91. Like the balsam of Peru, a colourless Tolu balsam is also now available.

An oil described as bois d'olho, olho vehermilho or oleo vermelho has been distilled from the wood of *Myrospermum erythroxylum* Allem. (probably a synonym of *Myroxylon peruriferum* Linn. f.), a Japanese tree which is also found in Brazil. This is a light-yellow mobile oil having an odour recalling that of a mixture of sandal, cedar and rose, and is obtained in a yield of 1 per cent. The oil has a use as a fixative in soap industry (Anon., 1925. *Perfum. essent. Oil Rec.*, 16 : 117, 163). It contains a high percentage of nerolidol (Naves 1948. *Perfum. essent. Oil Rec.*, 39 : 280).

Copaiba, known also as copaiba balsam or copaiva, is the oleoresin obtained from *Copaifera officinalis* Linn., *C. lansdorfii* Desf. and other species of *Copaifera* of tropical S. America. Its chemical and physical characters differ slightly according to the source, but a large percentage of volatile oil is always present. The Maracaibo variety from *C. officinalis* is a viscid, somewhat brownish-yellow and slightly fluorescent liquid, yielding 35 to 55 per cent. of volatile oil, while the Para variety from *C. lansdorfii* is a thinner, lighter yellow liquid, yielding up to 80 per cent. of volatile oil. The essential oil consists chiefly of sesquiterpenes among which caryophyllene is present. Copaiba oleoresin has not a wide application in perfumery, but it is an excellent fixative for perfumes used in the soap industry. In medicine it is used as a disinfectant, laxative, diuretic, and mild stimulant.

The African copaiba or illurin balsam (also known as hardwickia balsam) is a very fragrant oleoresin with a thick, pungent, pepper-like odour, and is obtained from a W. African tree, *Daniella thurifera* F.F. Benn. (Sierra Leone frankincense). It con-

tains 40 to 55 per cent. of an essential oil which is sometimes used as an adulterant of other oils. An oleoresin similar to illurin balsam is obtained from *D. oliveri* (Rolfe) Hutch. & Dalz. This oleoresin, under the name of wood oil, is one of the chief products of Nigeria.

The white, aromatic flowers of *Robinia pseud-acacia* Linn. (false acacia, black locust) of N. America, on solvent extraction, yield the robinia oil. This oil is dark coloured, and contains methyl anthranilate, indole, linalool, heliotropin, etc. (Elze, 1910. *Chem. Z.*, **34**, 814). The oil has a typical acacia odour.

Oil of supa is derived from *Sindora wallichii* Benth. of the Philippines. It consists of a mixture of sesquiterpenes in which cadinene is the chief constituent (Finne-more).

The fruits of the foreign ornamental shrub *Amorpha fruticosa* Linn. yield 1.1 to 3.5 per cent., and the leaves 0.5 to 0.8 per cent. of essential oils. The fruit oil has an odour similar to that of pepper oil. Its constants and constituents have been given by Finne-more.

From the roots of the foreign *Ononis spinosa* Linn. a small quantity (0.0066 per cent.) of an essential oil, known as cammock oil, has been reported (Haensel, 1910. *Apothekerztg.*, Berl., **25**: 303), but the Himalayan undershrub *O. hircina* Jacq. has not been investigated so far.

The following genera containing aromatic species have been described in detail later. They are distributed among the three sub-families of Leguminosae as under :—

Subfamily I. Papilionatae. *Dalbergia*, *Indigofera*, *Lathyrus*, *Melilotus*, *Psoralea*, *Trifolium*, and *Trigonella*.

Subfamily II. Caesalpinioideae. *Caesalpinia*, *Cassia*, and *Kingiodendron*.

Subfamily III. Mimosoideae. *Acacia*.

In addition to the plants belonging to the above eleven genera, a number of other plants are also aromatic. *Crotalaria occulta* R. Grah., a small undershrub of Assam, has terminal racemes of flowers with a delightful scent of violets; the corolla is sky blue with darker blue or purple veins. It is well worth cultivating in gardens. The asoka (*Saraca indica* Linn.), a middle-sized handsome tree found in different parts of India both in a wild state as well as under cultivation, bears orange-scarlet, fragrant flowers 1 to 1½ in. in length. The black siris [*Albizzia odoratissima* (Linn. f.) Benth.]

is a large deciduous tree of the Sub-Himalayan tract, Bengal, Central, Western and South India, with few-flowered heads of sweet-scented, white flowers. The kokko or siris [*Albizzia lebbbeck* (Linn.) Benth.], a large deciduous tree of the Sub-Himalayan tract, Bengal, Central and South India, which is also often planted as a roadside tree in various parts of the country, bears greenish-white fragrant flowers in pedunculate heads. The *kalkora* [*Albizzia kalkora* (Roxb.) Prain] of Assam is a large tree bearing heads of white flowers which are delightfully scented. The pink siris (*Albizzia mollis* Boiv., syn. *A. julibrissin* Durazz., var. *mollis* Benth.), a medium-sized tree of the outer Himalayas, bears heads of fragrant flowers. It is extremely handsome at the time of flowering, with innumerable pink tassels of delicate silky blossoms.

The heartwood of *karanj* [*Pongamia pinnata* (Linn.) Merr., syn. *P. glabra* Vent.], a moderate-sized tree found in many parts of India, both wild and planted, has a disagreeable odour when first exposed. The wood of the Burmese *Millettia leucantha* Kurz (*M. pendula* Benth. ex Baker) has a faint odour of tar.

The red sandalwood or sanderswood from *Pterocarpus santalinus* Linn. f., unlike as its name suggests, is nearly inodorous. Its powder, however, has been used as a cosmetic in India either in place of or with sandalwood to obtain a rose tint.

*Pterocarpus indicus* Willd. (Fl. Brit. Ind., in part, syn. *P. pallidus* Blanco) of Burma and the Philippines, bears sweet-scented flowers. From a specimen of its wood a solid aromatic substance, m.p. 70°, has been obtained by steam distillation (Anon., 1913, October. *Ber. Schimmel u. Co. Lpz.*: p. 68).

Among the constituents of essential oils reported from various members of the family may be mentioned: heptacosane, triacontane, unknown paraffin, styrene (styrol, vinylbenzene), phellandrenes, 'tolene' (C<sub>10</sub>H<sub>18</sub>), cadinene, caryophyllenes, copaene, unknown sesquiterpenes, azulene, benzyl alcohol, geraniol, nerol, linalool, farnesol, nerolidol, α-terpineol, sesquiterpene alcohol, phenol, vanillin, heliotropin, unknown aldehydes, unknown ketones, palmitic acid, cinnamic acid, methyl anthranilate, benzyl benzoate, benzyl cinnamate, coumarin, hydro-coumarin (melilotin, melilotol), cineole, and indole.

## 1. ACACIA (Tourn.) Linn.

(From the Greek *akakia*; originally a thorny tree found in Egypt.)

This genus comprises about 550 species of trees or erect or climbing shrubs. The leaf-bearing groups are scattered over the tropics and subtropics of the whole world, and the great phyllodineous series is almost restricted to Australia. In India it is represented by about 20 indigenous species as well as several introduced ones.

The acacias are important economically, not only for the timber of some species, but also for ornamental purposes and as the source of tanbarks, gums, and essential oils. Many of them are small trees and furnish excellent hard wood, which is serviceable for tool handles and for other small objects. The wood of the S. African *A. giraffae* Willd. is said to be extraordinarily hard. A few species like the Hawaiian *A. koa* A. Gray grow to a fairly large size, so much so that the Hawaiians formerly converted its trunks into dug-out war-canoes. The Australian blackwood (*A. melanoxylon* R. Br.) is used for cabinet work, coach-building, railway carriages, and agricultural implements, but the wood from the Indian-grown trees is said to be not so good as that from the Australian-grown plants. Most acacias are, however, used as firewood.

The bark of babul [*A. arabica* (Lam.) Willd.] has till recently been one of the premier tanning materials in India, and thousands of tons were being used annually by the tanneries in the country. It is, however, being ousted, in big tanneries at least, by the wattle bark from *A. mollissima* Willd. (black wattle), *A. decurrens* Willd. (green wattle), and related species, which is imported from S. Africa. Wattles are indigenous to Australia, but have been introduced into other countries, particularly Natal, S. Africa, and S. India. *A. pycnantha* Benth. (golden wattle) is much grown in Australia, but not in S. Africa. In Australia *A. mollissima* and *A. pycnantha* are of primary importance, while in S. Africa and in India *A. mollissima* and *A. decurrens* are the leading wattles. Of lesser importance as a tanning material is the bark of *A. dealbata* Link (silver wattle) in these countries.

Saponins are found in a number of acacias and a considerable trade exists in the saponaceous pods of *shikekai* [*A. concinna* (Willd.) DC.] which has already been dealt with.

A number of species yield gums, but the gum arabic from *A. senegal* (Linn.) Willd. and gum babul from *A. arabica* (Lam.) Willd. are commercial products which enjoy considerable trade. The best forms of these gums are colourless, odourless and tasteless.

The manufacture of *katha* and catechu from *A. catechu* (Linn.) Willd. (khair) and *A. chundra* Willd. (*lal-khair*) is an important industry in India, giving employment to hundreds of thousands of people. These products have already been referred to in the account of the family.

The leaves of Australian *A. dodonaeifolia* Willd. and *A. verniciflua* A. Cunn. are coated with resins.

The cassie perfume of commerce is obtained from the flower-heads of *A. farnesiana* (Linn.) Willd., while the flower-heads of *A. dealbata* Link and of some other species are used for the preparation of the well-known mimosa perfume. Both these are described in detail later.

*A. cavenia* Hook. & Arn. (Roman cassie, cassie Romaine) is cultivated in S. France for extraction of a perfume from its flowers. This species demands less care and yields a larger harvest of flowers than *A. farnesiana*, but the perfume is not so suave and well blended as that of cassie flowers. The flowers of Roman cassie are, for this reason, about half the price of the cassie flowers. Very little pomade is made from these flowers, but a concrete is prepared by extraction in petroleum ether. The yield of concrete varies from 0.60 to 0.84 per cent. (Naves & Mazuyer, 1947. *Natural Perfume Materials*: p. 165). The essential oil from flowers contains 40 to 50 per cent. of eugenol, 8 per cent. of methyl salicylate, and 42 to 52 per cent. of substances insoluble in dilute aqueous sodium hydroxide, consisting of benzyl alcohol (about 20 per cent.), geraniol, anisaldehyde, and eugenol methyl ether. Linalool, decyl aldehyde, and a ketone with an odour of violets, ionone or irone, are also probably present but could not be identified with certainty. The oil does not contain methyl anthranilate [Walbaum, 1903. *J. prakt. Chem.*, **68**: 235; vide *J. chem. Soc. (Abstr.)*; 1903, **84**(1): 845]. A distillate described by Naves, Sabetay & Palfray (1937. *Perfum. essent. Oil Rec.*, **28**: 336) had these characteristics: sp. gr. <sup>15°</sup> 1.031, [α] inactive, *n*<sub>D</sub><sup>20°</sup> 1.512, acid val. 11.2, and ester val. 96.0. Another set of readings was as follows: sp. gr. <sup>15°</sup> 1.028, [α] 0.1°, *n*<sub>D</sub><sup>20°</sup> 1.514, acid val. 22.0, and ester val. 112.0.



A small quantity of perfume is obtained for local use in Australia from the flowers of various species of *Acacia*, commonly known as wattle blossoms.

*A. jacquemontii* Benth., a bushy, thorny shrub of the dry regions of the Punjab, Sind, Rajputana and Gujarat, extending to the Suleman Range and lower Himalayas west of the Jhelum (usually along watercourses and in ravines), bears yellow, fragrant flowers in globose heads which are  $\frac{1}{3}$  inch in diameter. No essential oil has, however, been extracted from the flowers.

### 1. *Acacia dealbata* Link

Fl. Brit. Ind., II, 292 (without description).

(Silver Wattle, Mimosa)

This is a small to middle-sized, unarmed, evergreen Australian tree which was introduced into India before 1840. It bears grey foliage and bright-yellow, very sweet-scented flowers in globular beads about  $\frac{1}{3}$  inch in diameter. It has been tried in many places in the Himalayas, such as Abbottabad in the N.-W.F. Province, Almora in Kumaon, and Shillong in Assam, but nowhere has it succeeded so well as in the Nilgiris and Pulneys, where it has become completely naturalized.

The wood is used as a fuel. The bark, which contains 9 to 17 per cent. tannins, is a useful tanning material.

The flowers of this plant are used for the preparation of the well-known mimosa perfume, more or less in the same way as those of the next species *A. farnesiana* (Linn.) Willd. for the cassie perfume. Although *A. dealbata* is the principal species from which mimosa perfume is prepared, there are many others which are commercially known as mimosa. According to Poucher, "there are over 30 varieties cultivated on the Cote d'Azur and the cut branches are sold principally as cut flowers. The trade is so extensive that a special train leaves Mentone daily in January and February, and conveys to Paris large quantities of this favourite flower. Consignments average about 600,000 baskets of flowers each season... Visitors to the Riviera in February will have been charmed with the delicious fragrance and picturesque appearance of the mimosa trees as the train winds its way along the red rocky coast between Saint-Raphael and Cannes."

The next in importance to the flower-heads of *A. dealbata*, from the point of view of perfumery, are the flower-heads of *A. floribunda* Willd. The perfume of mimosa is faint (in small bunches) but captivating. In the case of *A. floribunda* it resembles cassie, while in the case of *A. dealbata* it is slightly coarser and resembles ylang-ylang. If several bunches were placed in a closed room, their fragrance is overpowering.

The first flowers from the middle of January to the end of March are sold as cut flowers in France. It is after this period, in April, when the trees flower profusely and the blossoms are more open and highly scented, that the flowers are collected and extracted for perfumery. In some years as much as 180,000 lbs. of flowers are collected for this purpose. They are then extracted with petroleum ether, since distillation and maceration yield inferior products.

The yield of concrete varies from 0.70 to 0.88 per cent., although in specially sorted flowers even 1.06 per cent. has been obtained. From this 20 to 25 per cent. of absolute is obtained. In humid season the yield of absolute is lower (Naves & Mazuyer, 1947. *Natural Perfume Materials*: p. 219).

The absolutes from both *A. dealbata* and *A. floribunda* are comparatively cheap and give excellent results in floral Colognes, while in de luxe perfumes, particularly honeysuckle and heliotrope, they give floral notes which are unique. Very fine bouquets of honey-like odour can be obtained by combination with muguet (lily of the valley) or jasmine (Poucher).

Very little is known about the chemistry of the oil, but von Soden (1925. *J. prakt. Chem.*, 110: 273) distilled with steam an absolute prepared by volatile solvents, and, calculated on fresh flowers, obtained 0.018 per cent. of a yellowish-green oil, which solidified towards 0° to a flaky crystalline mass. Its constants were: sp. gr. <sup>15°</sup> 0.816,  $[\alpha]$  inactive or slightly laevorotatory, acid val. 12, and ester val. 20.5.

A distillate described by Naves, Sabetay & Palfray (1937. *Perfum. essent. Oil Rec.*, 28: 336) had the characteristics:  $[\alpha]$  +0.3°,  $n^{20°}$  1.4812, acid val. 3.6, and ester val. 22.0.

A number of commercial mimosa oils are prepared synthetically without any floral extract. These artificial perfumes, which resemble cassie, are compounded from methylacetophenone and its homologues, together



FIG. 33 — *Acacia farnesiana* (Linn.) Willd.

with methyl heptine carbonate, terpineol, and hydroxycitronellal. Although good results are obtained by this method, the addition of 10 per cent. of mimosa absolute makes all the difference to the smoothness and fixity of the blended mixture.

## 2. *Acacia farnesiana* (Linn.) Willd.

Fl. Brit. Ind., II, 292.

(Cassie Flower, *Vilayati-babul*)

This is a spiny shrub, or low tree with slender, zigzag branches marked with grey dots. It bears during the cold season bright-yellow, very fragrant flowers in globose heads  $\frac{2}{3}$  to  $\frac{1}{2}$  inch in diameter, which per-

fume the surrounding atmosphere very pleasantly. A native of tropical America, this plant is now widely cultivated all over India and in the East, especially as a hedge plant. It is also found in Egypt, Syria, Algeria and France, where it is specially cultivated for the industrial extraction of perfume.

The stem yields a gum which is said to be collected and marketed in Sind and the Punjab, especially as an adulterant of gum babul. The bark and the pods contain tannins.

The round, yellow flower-heads constitute the cassie flowers (known in France as *cassie ancienne*) which are used in European perfumery. Their odour closely resembles

a perfectly blended combination of orange blossom and violet with just a suggestion of cumin.

In France it is commercially cultivated in Provence on the outskirts of Cannes, Cannet, Vallauris, and Saint-Laurent-du-Var. It thrives best on a light sandy granite soil. The plants are raised from seeds and begin to bear flowers after 3 years. They blossom from the end of September to the beginning of February, but the main collection is usually made during September to November. The flowers collected during October are the most valuable. In the spring season the trees are trimmed.

The flowers are collected twice a week, one by one, during the day time and are conveyed to the works in the evening. The blooming is successive, some flowers being ready for collection before the others are scarcely formed. The total yield per tree in one year varies from 4 to 10 lbs.

In Algeria and Egypt also the principal harvest is collected during October and November.

The perfume of cassie flowers is very delicate and is destroyed if distilled in steam. The flowers are, therefore, extracted by maceration in fats at 35° to 50°C., or by petroleum ether. The absolute is prepared from both pomade and concrete.

The yield of concrete generally varies between 0.50 and 0.70 per cent. The flowers gathered at the end of the season usually give a higher yield than those of the autumn. The concrete yields about 30 per cent. of absolute, which furnishes 6.5 to 9 per cent. of steam-distillable products. It is of a soapy consistency, dark brown in colour, becoming lighter on contact with light and air. The distillate is colourless and very refractive (Naves & Mazuyer, 1947. *Natural Perfume Materials* : p. 166).

Walbaum & Rosenthal (1929. *Ber. Schimmel u. Co. Lpz.* : p. 193) have observed a concrete having the following characteristics : congealing point 46.5° and saponification val. 103.6.

Naves & Mazuyer (*loc. cit.*) mention the following characteristics of an absolute of Grasse origin : sp. gr. <sup>15°</sup> 0.988, acid val. 62.8, and ester val. 33.5.

Rovesti (1925. *Profumi ital.*, 3 : 277) gives the following constants for an absolute prepared in Liguria : sp. gr. <sup>15°</sup> 1.020 to 1.070, [ $\alpha$ ] 0° to -3°, *n* 1.514 to 1.521, acid val. 18 to 55, and ester val. 97 to 243.

Naves & Mazuyer (*loc. cit.*) give the following analytical data for the products obtained by steam distillation. These have been compiled from the works of von Soden (1904. *J. prakt. Chem.*, 69 : 270), Schimmel & Co. (1907, April. *Ber. Schimmel u. Co. Lpz.* : p. 18), and Naves, Sabetay & Palfray (1937. *Perfum. essent. Oil Rec.*, 28 : 336).

The distillate from the pomade concentrate prepared in India was obtained by Schimmel & Co. (1904, April. *Ber. Schimmel u. Co. Lpz.* : p. 21), with a yield of 0.171 per cent., based on pomade. This distillate had a pale-yellow colour, with the following characteristics : sp. gr. <sup>15°</sup> 1.0475, [ $\alpha$ ] 0°, *n*<sup>20</sup> 1.5133, and saponification val. 176.0.

Schimmel & Co. (1899, Oct. *Ann. Rep.* : p. 58 ; 1901, Apr. *ibid.* : p. 18 ; 1903, Apr. *ibid.* : p. 17) report an Indian cassie pomade to contain 11 per cent. of methyl salicylate, small amounts of *p*-cresol, benzaldehyde, benzyl alcohol, a ketone possibly menthone, anisaldehyde, decyl aldehyde, cuminaldehyde, and possibly geraniol and linalool. A constituent of importance as far as odour is concerned is a ketone of violet odour and unknown constitution. Farnesol has also been reported (Haarmann & Reimer, *vide* Wehmer).

In practical perfumery cassie absolute is used in the preparation of numerous violet bouquets to which it imparts a delightful and peculiar aroma unobtainable with any other product. Owing to the high cost, it is not possible to add natural violet absolute when compounding synthetic violet ottos. Cassie absolute in these cases serves as an excellent cheap substitute, and about 5 per cent. of it may be used with satisfactory results.

ORIGIN AND % YIELD			CONGEALING POINT	SP. GR. <sup>15°</sup>	OPTICAL ROTATION	REFRACTIVE INDEX <sup>20°</sup>	ACID VAL.	SAPONIFICA TION VAL.
France :	0.084 of flowers	...	18-10	1.040(27°)	0.4°	—	42.50	114.0
	5.65 of concrete	...	liq.	1.0575	0.3°	1.5158	25.40	229.0
Egypt }	...	...	liq.	1.032	0.6°	1.5045	5.60	154.0
Egypt }	6.5 to 9 of concrete	...	liq.	1.037	0.2°	1.5082	3.70	148.0
Egypt }	...	...	liq.	1.029	1.1°	1.5018	4.20	166.0
Egypt }	...	...	liq.	1.043	0.2°	1.5120	6.30	182.0
Egypt :	26.4 of absolute*	...	liq.	—	inactive	1.5112	30.50	169.0

\*Prepared from a commercial product of guaranteed purity

FIG. 34 — *Caesalpinia sappan* Linn.

On the basis of constituents given above synthetic cassie oil is manufactured, and rounded off with a judicious addition of concrete of natural flowers.

It is said that a good deal of pomade was at one time made in India, perhaps in the Punjab and to a certain extent in the United Provinces, but for some unknown reason this industry has died. Efforts should be made to revive this since cassie perfumes command, even today, a good market.

## 2. CAESALPINIA Linn.

(In honour of Andreas *Caesalpini*, professor of medicine at Pisa in the sixteenth century. He was the first botanist to classify plants by the flowers and fruits.)

This genus comprises about 60 species of tropical and subtropical trees, shrubs or

often prickly climbers, out of which 12 are represented in India.

The trees have a hard wood, which in several species yields a dye. The sappan-wood (*C. sappan* Linn.) yields the well-known red dye which was in considerable use formerly. Other important dyewoods are *C. crista* Linn. (redwood), *C. brasiliensis* Linn. (Brazil wood), and *C. echinata* Linn. (peachwood), all of America.

The barks and pods are usually rich in tannin. The pods of divi-divi or American sumach [*C. coriaria* (Jacq.) Willd.], which is often cultivated in India, are a valuable tanning material and contain 40 to 50 per cent. of tannin. The pods of *teri* (*C. digyna* Rottl.) are also one of the richest sources of tannin, and are used in India for tanning purposes.

Some species are used in medicine, and contain a resin.

The peacock flower or Barbados pride [*C. pulcherrima* (Linn.) Sw.] is a handsome shrub with scarlet or yellow flowers. It is commonly cultivated in Indian gardens as an ornamental plant.

*C. nuga* (Linn.) Ait., a large prickly climbing shrub of the coastal forests of India, bears yellow, fragrant flowers in long-peduncled terminal racemes 10 to 15 inches in length.

### **Caesalpinia sappan** Linn.

Fl. Brit. Ind., II, 255.

(Sappanwood, *Patang*)

This is a small prickly tree with panicles of yellow flowers  $\frac{3}{4}$  inch in diameter, and obliquely oblong, 3- to 4-seeded, woody pod having a short curved beak at the upper angle of the obtuse apex. It is found in Bengal, S. India and Bombay, usually cultivated.

The dark-red or orange-yellow wood yields a valuable red dye which was in considerable use formerly, and even exported. This dye, which is still used to an extent, is soluble in water, and is employed in dyeing wool and in calico-printing.

The leaves, on distillation, yield 0.16 to 0.25 per cent. of an almost colourless oil having sp. gr.  $25^{\circ}$  0.825 and  $[\alpha] +37.3^{\circ}$  to  $+50.3^{\circ}$ . The odour of the oil is pepper-like, which appears to consist chiefly of *d*- $\alpha$ -phellandrene. The bulk of the oil boils at  $170^{\circ}\text{C}$ . (Gildemeister). An olefinic hydrocarbon is also reported (Romburgh, 1925, *vide Chem. Abstr.*, 1926, 20 : 2722).

### 3. **CASSIA** Tourn. *ex* Linn.

(The classical name of some trees with aromatic bark.)

This genus comprises 400 species of trees or shrubs, rarely herbs, of tropical and warm temperate regions, and is represented in India by 15 species. Its members have abruptly pinnate leaves and large, showy, nearly regular, usually yellow flowers in racemes or clusters.

Several species are extensively cultivated throughout the tropics for the elegance of their foliage combined with the beauty of their flowers. Among these may be mentioned the arboreous *C. fistula* Linn. and *C. renigera* Wall., and the shrubby *C. bicapsularis* Linn., *C. tomentosa* Linn. *f.*, and *C. laevigata* Willd.

A number of species are of considerable importance from the medicinal point of view, and some are widely used for their cathartic properties. The senna leaves and pods, which are widely used as purgatives, are derived from the Indian or Tinnevely senna (*C. angustifolia* Vahl) and the Alexandrian senna (*C. acutifolia* Delile). The pulp of the fruit of *C. fistula* Linn. (Indian laburnum, purging cassia) is one of the commonest domestic medicines, and acts as a simple purgative. The purging property of cassias depends upon chrysophanic acid and other oxyanthraquinone derivatives which they contain. Some species, such as the ringworm shrub (*C. alata* Linn.), are used in skin diseases.

The bark of a number of species contains a fair quantity of tannin. That of *C. fistula* is commonly used in India for tanning purposes under the name of *sonari* bark.

The seeds of some species are used by the poor people as a substitute for coffee.

From the point of view of essential oils, this genus has no economic importance. The leaves of *C. angustifolia* are said to have a tea-like odour and to contain traces of an essential oil (Tschirch, 1898. *Ber. dtsh. pharm. Ges.* : p. 189). The seeds of *C. occidentalis* Linn. (*kasondi*), when roasted, emit an aroma similar to that of coffee; they are known as negro coffee. The leaves of *C. tora* Linn. (*chakunda*) have a foetid smell, but the seeds on roasting develop a coffee-like aroma. Both these species are common weeds of roadsides, river banks, and fallow lands in most parts of India.

### 4. **DALBERGIA** Linn. *f.*

(In honour of Nicholas Dalberg, a Swedish botanist, 1730-1820.)

This genus comprises over 200 species of tropical trees, shrubs, and woody climbers, out of which about 20 are found in India. Its members bear copious, papilionaceous flowers in terminal or lateral panicles, and oblong or strap-shaped, thin and flat, indehiscent, 1- to 4-seeded pods.

The genus is very important in Indian forest economy, as it contains two of the most valuable and important Indian forest trees, besides several others of interest on account of their woods and other products or their value in silviculture. The Indian *D. sissoo* Roxb. (*sissoo*, *shisham*) and rosewood or Bombay blackwood (*D. latifolia* Roxb.) enjoy considerable trade. *D. latifolia*, to-

gether with the Burmese species *D. cultrata* R. Grah. ex Benth. (Burma blackwood) and *D. oliveri* Gamble ex Prain (Burma tulipwood, *tamalan*), is of great ornamental value and is, therefore, of interest to connoisseurs of fancy woods: *D. sissoo* produces a valuable timber which seasons well and is esteemed for all purposes where strength and elasticity are required. *D. melanoxylon* Guill. & Perr. is the African blackwood or Senegal ebony of the trade, and *D. nigra* Allem ex Benth. is the Brazilian rosewood. Cocobolo is derived from the American *D. retusa* Hemsl. and related species, and is one of the showiest and most strikingly coloured woods. The heartwood is orange to orange red in colour, streaked with jet black. It is very hard, tough and strong, and is largely used in cutlery. The dye in cocobolo appears to be allied to that of *Acacia catechu* (Linn.) Willd.

The highly ornamental heartwood of the Indian *D. latifolia* has a fragrant scent. Similarly the heartwood of the Burmese *D. oliveri* has a faint, pleasant odour.

*D. rimosa* Roxb., a suberect or climbing often tendril-bearing shrub of the E. Himalayas and Assam, bears panicles of sweet-scented, very small flowers (among the smallest in Papilionatae).

The scented wood of *D. cumingiana* Benth., of the Philippines and the Dutch East Indies, yields about 0.5 per cent. of an essential oil with aromatic odour. The oil has the following characteristics: sp. gr.  $^{26^{\circ}}$  0.891,  $[\alpha]$  -4.3°, ester val. 5, and the same after acetylation 116 (Parry).

The heartwood of the thick stems near the ground and of the roots of the climbing *D. parviflora* Roxb.\* of Malay Peninsula and Sunda Islands is scented and exported to China for the manufacture of joss sticks. It is used in Borneo and Celebes as an incense. According to Prain (1895. *Ann. R. bot. Gdn. Calcutta*, 10: 35), this is the *kayu-laka* of commerce. Spoelstra (1931. *Rec. Trav. chim. Pays-Bas*, 50: 433) has obtained an essential oil from the wood in a yield of 0.45 to 0.8 per cent., with *l*-nerolidol ( $C_{15}H_{26}O$ ) as the main constituent. According to him traces of furfural and probably farnesol are also present. The oil has the following characteristics: sp. gr.  $^{15^{\circ}}$  0.8878 to 0.8929,  $n_D^{20}$  1.4809 to 1.4825,  $[\alpha]$  -0.20° to -4.75°, acid val. 0.5 to 1.6, ester val. 0 to 1.2, and the same after acetylation 139.5 (=61.6 per cent.).

\*This plant is probably also found in the Andamans.

## 5. INDIGOFERA Linn.

(From the Latin *indicum* — a blue pigment believed to be indigo, and *fero* — I bear; species of this genus yield the indigo of commerce.)

This genus comprises about 350 species of herbs or shrubs of warm regions. In India it is represented by 40 species having odd-pinnate leaves and pink or purple papilionaceous flowers in axillary racemes.

The most famous of its members is *I. tinctoria* Linn. which furnishes indigo.

The seeds of several species are said to be poisonous.

### *Indigofera galegoides* DC.

Fl. Brit. Ind., II, 100.

This is a tall shrub about 12 ft. high, with twiggy branches and pale-red flowers in dense racemes 2 to 3 inches in length. It is found in the Khasi Hills at an altitude of about 5,000 ft., generally in shady localities.

The fresh plant, mashed with water, yields on distillation 0.2 per cent. of a light-yellow essential oil. This oil has an odour of bitter almonds, but differs from the latter in having a herbaceous aroma. Its sp. gr. is 1.046. It contains benzaldehyde, hydrocyanic acid, and small quantities of ethyl and methyl alcohol (Anon., 1894, Oct. *Ber. Schimmel u. Co. Lpz.*: p. 75; Anon., 1896, Apr. *ibid.*: p. 75).

## 6. KINGIODENDRON Harms

This genus comprises two species of which one is found in India.

### *Kingiodendron pinnatum* (Roxb.) Harms

*Hardwickia pinnata* Roxb., Fl. Brit. Ind., II, 270.

(Piney, *Shurali*)

This is a very large, handsome tree having leathery leaflets and numerous, very small white flowers in panicles of racemes. It is found in the evergreen forests of W. Ghats from S. Kanara to Travancore.

The wood is dark red or reddish brown, and is locally used for building purposes as well as for making furniture.

The tree yields a viscous, dark reddish-brown balsam with not an unpleasant odour. In smell and taste, this resembles copaiba balsam which is derived from the

foreign *Copaifera officianalis* Linn. and allied species. The balsam is used in indigenous medicine, and also as a varnish for wood after thinning with turpentine. It is obtained from the tree trunk by tapping, for which purpose large trees with a girth of 5 to 6 ft. are selected. A notch is cut in the trunk, into which a tube or a trough is inserted to carry the exudation to a receptacle at the base of the tree. The balsam begins to drain at once, but the flow stops in about four days. This operation is performed when the rains have set in, since during this period the yield is more. In some places the trees are tapped in the dry season from December to May. A healthy tree of 8 ft. girth is reported to yield as much as 12 gallons of the balsam.

According to Weigel (1906. *Pharm. Zentralh.*, 47: 773) kingiodendron balsam is analogous to copaiba balsam. Hooper (1907. *Pharm. J.*, 78: 4), who examined two genuine Indian samples, gives a resumé of the work done on the balsam by various workers and concludes that the physical constants easily distinguish kingiodendron balsam from the copaiba and gurjun balsams. Iyer & Sudborough (1918. *J. Indian Inst. Sci.*, 2: 29), who examined two samples from Travancore and Mysore, are of the opinion that kingiodendron balsam resembles copaiba balsam more closely than it does gurjun balsam. The following are the characteristics of the balsam as recorded by Iyer & Sudborough; Hooper's values are also given:—

	TRAVANCORE SPECIMEN	MYSCORE SPECIMEN	HOOPER'S VALUES
Specific gravity <sup>15°</sup> ...	1.008	0.997	1.007-1.012
Acid val. ...	90.2	84.6	97.2-99.8
Saponification val. ...	112.9	103.7	106-112
Acetyl val. ...	79.6	83.0	—
Per cent. of volatile oil ...	42.7	47.0	39.5-41.1

Schimmel & Co. (1905, Apr. *Ann. Rep.*: 1907, Apr. *ibid.*) by steam distilling the balsam obtained 44 per cent. of a colourless oil having the following constants: sp. gr. <sup>15°</sup> 0.9062,  $[\alpha]$  7.4°, acid val. 0.85, ester val. 2.88, and soluble in about 5 and more volumes of 90 per cent. alcohol.

Iyer & Sudborough (*loc. cit.*) obtained from 2 samples on steam distillation by superheated steam 42.7 and 47.0 per cent. of a practically colourless oil having a characteristic odour and a pungent bitter taste. The major portion of the oils distilled at 257° to 271°C. at ordinary pressure. They have recorded the following constants:

	TRAVANCORE OIL	MYSCORE OIL
Specific gravity <sup>15°</sup> ...	0.931	0.908
Refractive index <sup>20°</sup> ...	1.500	1.500
Optical rotation <sup>15°</sup> in 100 mm. tube ...	-1.72°	-7.86°
Acid val. ...	trace	trace
Saponification val. ...	nil	nil
Acetyl val. ...	12.6	1.4

These physical characteristics resemble those of caryophyllene, the main constituent of clove oil, but the kingiodendron essential oil needs further careful examination for caryophyllene with a view to discovering an alternative source of clove oil which is not produced in India.

Iyer & Sudborough (*loc. cit.*) have also examined the resin obtained after the distillation of the essential oil. The resin melts at 53° to 56°C., and has the following characteristics:

	TRAVANCORE RESIN	MYSCORE RESIN
Specific gravity ...	1.088	—
Acid val. ...	162.2	160.0
Saponification val. ...	193.9	200.0
Ash ...	0.05%	0.02%
Solubility in 90% alcohol ...	99.2%	complete

## 7. LATHYRUS Linn.

(From the Greek *lathyros*, a kind of pulse.)

This genus comprises 110 species of annual or perennial climbing herbs, which are distributed chiefly in the north temperate zone. Of the eight species found in India, *L. sativus* Linn. (*khesari*) is the most important, since it is largely cultivated all over the country as a cold-weather crop for use as a fodder and by the poorer classes of people as a food. The whole plant as well as the seeds, under certain circumstances, have been incriminated in the production of poisonous symptoms or the disease called lathyrism.

The sweet pea (*L. odoratus* Linn.) is cultivated in gardens for the sake of its various coloured, sweetly fragrant flowers. This is described below in detail.

### *Lathyrus odoratus* Linn.

(Sweet Pea)

This climbing herb is commonly cultivated in the Indian gardens for the sake of its elegant, various coloured flowers having a delicate and sweet fragrance, recalling that of orange flowers and hyacinth with a suggestion of rose.

The flowers contain a small amount of essential oil, but this does not appear to have been systematically investigated. It is also doubtful whether they have ever been extracted for use in perfumery, although there is reason to believe that eufleurage would capture the floral fragrance faithfully. The odour of the flowers suggests the presence of methyl anthranilate.

The sweet pea perfumes of commerce are synthetic and are generally compounded from methyl anthranilate, benzylideneacetone, hydroxycitronellal, and terpineol. Numerous other synthetic perfumes are employed, and are rounded off with natural essences, such as jasmine and bergamot.

## 8. MELILOTUS Tourn. ex Hall.

(From the Greek *meli* — honey, and *lotos* — lotus; the flowers are much visited by bees.)

This genus comprises 20 herbaceous species (sweet clovers) which are distributed over the temperate and subtropical regions. It is characterized by trifoliate leaves and slender racemes of yellow or white flowers succeeded by subglobose or oblong, indehiscent or tardily dehiscent pods.

The sweet clovers are a valuable fodder either in the green condition or when made into hay. They are also cultivated for pasture and for soil improvement. In India they are represented by three species, namely (1) *M. alba* Desr. the white-flowered sweet clover, (2) *M. indica* (Linn.) All. (*M. parviflora* Desf.) the pale-yellow-flowered sweet clover, and (3) *M. officinalis* Lam. the yellow-flowered sweet clover.

All species of *Melilotus* contain coumarin, which is concentrated mainly in mature and drying branches. Coumarin is one of the important perfumes, and is a basic material for all perfumes of the type of *foin-coupé* or "new-mown hay". It is found in the largest amount (3 per cent.) in tonka beans [*Dipteryx odorata* (Aubl.) Willd. and *D. oppositifolia* (Aubl.) Willd.]. Commercial coumarin is, however, invariably a synthetic product.

### 1. *Melilotus alba* Desr.

Fl. Brit. Ind., II, 89.

(White-flowered Sweet Clover)

This is a biennial herb very much like *M. officinalis* Lam., but bears white, scented flowers. It is found in the plains

of N. India, ascending up to an altitude of 12,000 to 13,000 ft.

The entire plant contains coumarin.

### 2. *Melilotus indica* (Linn.) All.

*M. parviflora* Desf., Fl. Brit. Ind., II, 89.

(Pale-yellow-flowered Sweet Clover, *Banmethi*, *Senji*)

This is a slender annual 1 to 2 ft. high with pale-yellow flowers in spicate racemes. It is found in Bombay, Bengal, the United Provinces, and the Punjab, either cultivated or as a weed of cultivation.

The plant is said to contain coumarin (Watt.).

### 3. *Melilotus officinalis* Lam.

Fl. Brit., Ind., II, 89.

(Yellow-flowered Sweet Clover, *Aspurk*)

This is a tall and robust annual or biennial herb bearing yellow, odorous flowers crowded in bunches 1 to 2 inches long. It is found at Nubra and in Ladakh at altitudes of 10,000 to 13,000 ft., and is occasionally cultivated in India as a fodder.

The entire plant contains coumarin (0.2% on dry basis) together with melilotic acid, coumaric acid and an oily substance. Hydrocoumarin has also been reported (Vogel, 1820, *vide* Wehmer; Zwenger, 1867. *Ann. Chim., Suppl.*, 5: 100; Phipson, 1875. *Chem. News*, B2: 25).

The flowers yield 0.0133 per cent. of an essential oil (melilot oil) containing coumarin (Haensel, 1900. *Apothekerztg, Berl.*, 15: 516). The seeds yield 7.83 to 8.35 per cent. of a fatty oil with coumarin smell.

## 9. PSORALEA Linn.

(From a Greek word *psoraleos*, meaning scabby.)

This genus comprises about 100 species of tropical and subtropical herbs and undershrubs having glandular compound leaves and spicate or racemose, purple or white flowers. It is represented in India by two species, *P. corylifolia* Linn. and *P. plicata* Delile. Besides these, the S. African shrub *P. pinnata* Linn., with blue flowers and narrow leaflets, has also established itself about Ootacamund.

The N. American *P. esculenta* Pursh (prairie turnip) has edible roots. The S. American *P. glandulosa* Linn. is locally used for tea.

The leaves of *P. bituminosa* Linn. of the Mediterranean region have an asphalt-like odour when crushed. They yield 0.048 per cent. of an essential oil, which is semi-solid at ordinary temperature. It has sp. gr. <sup>25°</sup> 0.8988, acid val. 57, and ester val. 12. Fatty acids, melting point 38° to 40°, have been isolated (Finnemore).

***Psoralea corylifolia* Linn.**

Fl. Brit. Ind., II, 103.

(Babchi)

This is an erect annual 1 to 3 ft. high, having firm branches copiously covered with conspicuous gland dots. It has gland-dotted leaves and bluish-purple flowers. The pods are small, black, 1-seeded. The seeds are aromatic with a pungent bitterish taste. The plant occurs in the plains throughout India, from the Himalayas to Ceylon.

The seeds have a persistent odour and are employed locally in the preparation of certain types of medicated oils and incense preparations. They are commonly used in indigenous medicine, particularly against cutaneous diseases, such as leucoderma and leprosy. Their essential oil has a powerful effect against the skin streptococci. Local applications of oleoresinous extracts from the seeds have recently been found to be beneficial in the treatment of leucoderma of non-syphilitic origin.

On distillation with steam, the powdered seeds yield 0.05 per cent. of a pale-yellow oil with a marked odour of the seeds. The petroleum ether extraction of the seeds gives a reddish-brown oil and crystalline solids psoralene and isopsoralene which are coumarone-coumarin compounds. The oil contains a considerable quantity of resin (Jois, Manjunath & Rao, 1933. *J. Indian chem. Soc.*, **10**: 41; Jois & Manjunath, 1936. *Ber. dtsch. chem. Ges.*, **69**: 399; Späth *et al.*, *Ber. dtsch. chem. Ges.*, **69**: 1087).

The leaves are gland dotted and may contain an essential oil like the foreign *P. bituminosa* Linn.

**10. TRIFOLIUM (Tourn.) Linn.**

(The Latin name for clover; referring to the three leaflets.)

This genus comprises about 300 species of herbs (clovers) having trifoliate leaves,

small red or white sometimes yellow flowers in dense axillary heads, spikes or umbels, and minute membranous, 1- or few-seeded indehiscent pods. Its members are found in the temperate and subtropical regions. It is represented in India by 5 species.

Clovers are important pasture and hay plants, although some of the species are believed sometimes to produce untoward symptoms in animals feeding on them. For example, although *T. pratense* Linn. (red clover or cow grass) and *T. hybridum* Linn. (alsike clover) are normally considered as valuable fodder plants, some authors refer to symptoms of "photosensitization" when taken by animals in large quantities. Similarly *T. repens* Linn. (Dutch clover, white clover), normally an excellent fodder, is said to contain in fresh plants a cyanogenetic glycoside and to produce poisonous symptoms in animals.

The blossoms of a number of clovers exhale a honey-like fragrance. Of the odorous species, *T. incarnatum* Linn. (carnation clover, scarlet clover) of Europe and *T. odoratum* Shrank (probably a synonym of *T. montanum* Linn.) of Italy are the best known. Rogerson (1910. *J. chem. Soc.*, **97**: 1004) has obtained from the flowers of *T. incarnatum* 0.029 per cent. (calculated on dry clovers) of a powerfully odorous pale-yellow essential oil having sp. gr. <sup>20°</sup> 0.9597 and  $[\alpha]_D -1.5^\circ$ . He has identified furfural as constituent of the oil.

The odour of clover is not extracted on a commercial scale, and the trèfle (trefoil) perfumes marketed by a number of perfumers are not the products of any species of this genus. On the other hand perfumes bearing the name trèfle are manufactured synthetically, and are based on a large proportion of amyl salicylate, together with isobutyl salicylate, phenylacetic acid, vanillin, benzylideneacetone, jonquil, ylang-ylang, tuberose, jasmine, etc.

***Trifolium pratense* Linn.**

Fl. Brit. Ind., II, 86.

(Cow Grass, Red Clover, Trèfle)

This is a perennial herb about a foot or more high with usually red-purple flowers in dense roundish heads  $\frac{3}{4}$  to  $1\frac{1}{2}$  inches in dia-



meter, and 1-seeded pods. It is found in the Himalayas from Kashmir to Garhwal at altitudes of 4,000 to 8,000 ft. It is one of the common forage clovers in these regions. It is regarded as a good cropper where the commoner clover fails.

A rather unpleasant-smelling oil containing furfural has been obtained in a yield of 0.028 per cent., calculated on dry flowers. The oil has sp. gr.<sup>20°</sup> 0.9476 and  $[\alpha] + 4.0^\circ$  (Power & Salway, 1910. *J. chem. Soc.*, **97**: 231).

## 11. TRIGONELLA Linn.

(From the Greek *treis* — three, and *gonia* — an angle; referring to the triangular form of the flowers.)

This genus comprises 70 herbaceous species out of which 8 are found in India. It is found in the Mediterranean region, Europe, Asia, S. Africa, and Australia. Its representatives are fragrant annuals having trifoliate leaves, small, yellow or white flowers in heads or racemes, and linear many-seeded pods.

## *Trigonella foenum-graecum* Linn.

Fl. Brit. Ind., **II**, 87.

(Fenugreek, *Methi*)

This strongly and pleasantly scented annual is widely cultivated, particularly in N. India, for the sake of its leaves and seeds.

The green leaves are eaten as a potherb, and the dried ones as a flavouring agent. The seeds are used in medicine and as a spice.

Fenugreek is locally used as an insect repellent. The agriculturists in the Kangra district in the Punjab mix the dried plant with their grains stored up in bags, in order to protect the cereals from attacks of insects during the rainy season.

In Java the seeds are said to be used in cosmetics and hair oils.

The seeds contain the alkaloid trigonelline, choline, and 0.014 per cent. of a brown essential oil of intense odour. The oil has sp. gr.<sup>13°</sup> 0.870 and  $[\alpha] + 0.8^\circ$  in 10 per cent. alcoholic solution (Haensel, 1910. *Apothekerztg., Berl.*, **25**: 303).

The odoriferous principle of the leaves does not appear to have been examined.

*Supplement*

AROMATIC PLANTS OF INDIA



FIG. 35 — *Psoralea Corylifolia* Linn.

# AROMATIC PLANTS OF INDIA

## PART X

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### Family XLI—ROSACEAE

(Rose Family)

THIS is a large family comprising about 100 genera and over 2,000 species of herbs, shrubs, and trees which are spread over the whole world, but most abundant in north temperate regions.

Many plants of this family are cultivated for ornamental purposes; roses, spiraeas, flowering cherries, and plums are some of the prominent examples. A few only, such as *Prunus puddum* Roxb. ex Wall., yield timber of some value. There are, however, a large number of plants which furnish valuable fruits, such as the apricot (*Prunus armeniaca* Linn.), peach [*P. persica* (Linn.) Stokes], almond (*P. amygdalus* Batsch), sweet cherry or gean (*P. avium* Linn.), plums and prunes (*P. domestica* Linn.), *alucha* (*P. communis* Huds., var. *insititia* Hook. f.), cherries (*P. cerasus* Linn. and others), loquat [*Eriobotrya japonica* (Thunb.) Lindl.], strawberry (*Fragaria vesca* Linn.), raspberry and blackberry (*Rubus* sp.), apple (*Pyrus malus* Linn.), pears (*P. communis* Linn. and *P. sinensis* Lindl.), quince (*Cydonia oblonga* Mill., syn. *Pyrus cydonia* Linn. and *Cydonia vulgaris* Pers.), etc.

Some plants are used for medicinal purposes, and the root, bark, leaves, and flowers of these are said to possess astringent, tonic, and anthelmintic properties. The mucilaginous seeds of *Cydonia oblonga* have been used in indigenous medicine for a long time as a demulcent in the treatment of gastro-intestinal and other conditions.

Many of the plants of this family possess poisonous properties and these are due to the cyanogenetic glycosides, such as amygdalin, prunasin, etc., which occur in the leaves, bark, and seeds. Of these, amygdalin occurring in bitter almonds (*Prunus amygdalus* Batsch, var. *amara* of authors) is well known.

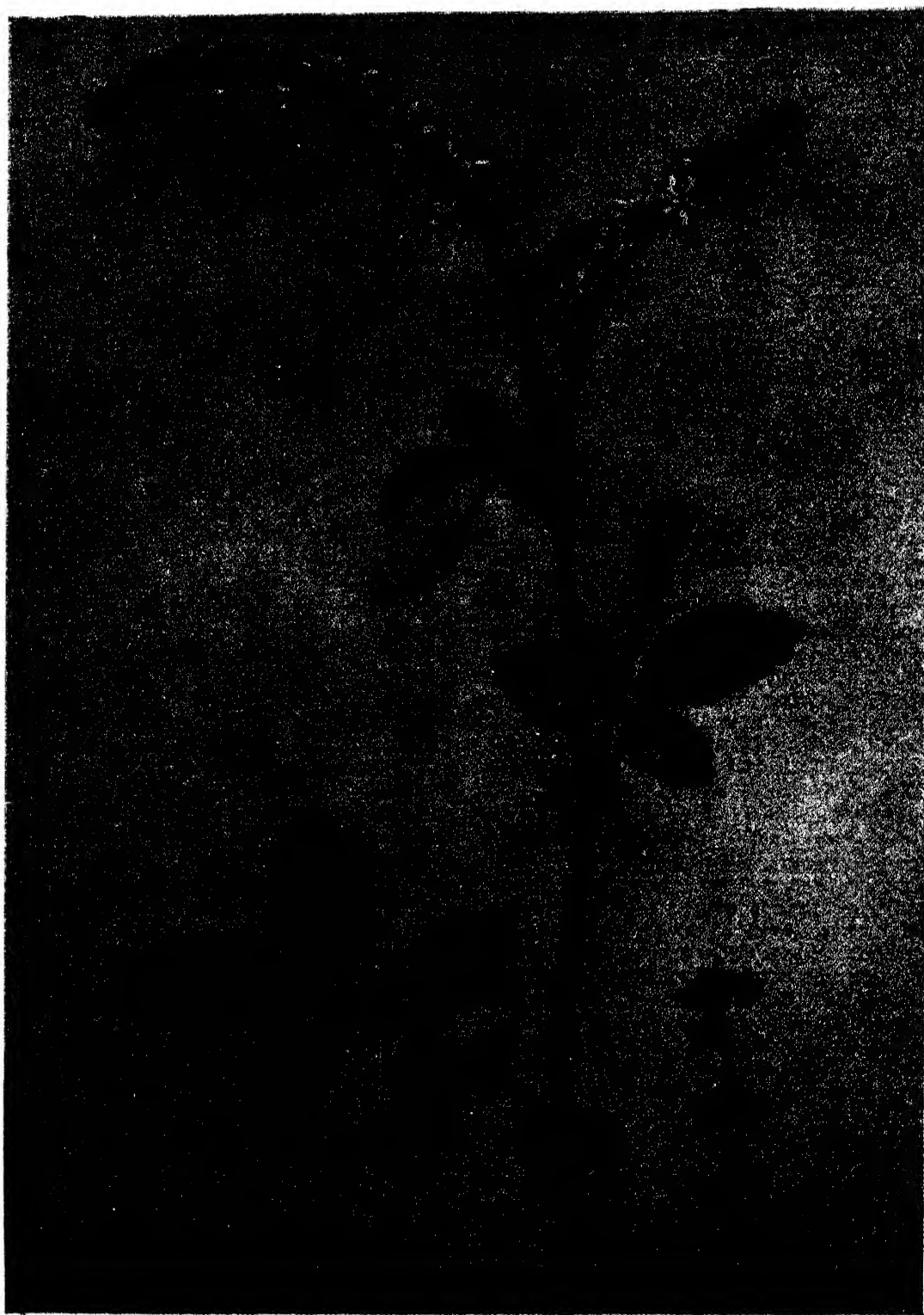
Apart from cyanogenetic glycosides, other glycosides, such as phloridzin, gaultherin, etc., saponins, such as those in the bark of *Quillaja saponaria* Molina (soapbark tree) of Chile, etc., and essential oils are found in plants of this family.

Almost all the fruits mentioned above and many others (such as those of species belonging to *Fragaria*) have a flavour and aroma peculiar to each, and this is due to the presence of some essential oil or the other, possibly as esters or aldehydes. None of these, however, is extracted because of the very small quantity present in expensive fruits. For this reason, primarily, the fruit essences are prepared from synthetic products, simulating the natural flavours.

Oil of roses, which has perhaps been the most persistently popular perfume from antiquity, is derived from flowers of certain species of *Rosa* which are cultivated for the purpose. Many other plants belonging to *Crataegus*, *Geum*, *Prunus*, *Pyrus*, *Rubus*, and *Spiraea* are also known to contain essential oils, and will be described under the accounts of these genera.

The flowers of *Filipendula ulmaria* Maxim. (*Spiraea ulmaria* Linn.) of Europe, N. America, and N. Asia yield 0.2 per cent. of an essential oil, which is heavier than water. The constituents of the oil are salicylaldehyde, methyl salicylate, and traces of heliotropin and vanillin (Wehmer). The salicylaldehyde is not found in the flowers as such, but is formed during distillation. The roots of this species also yield an oil the main constituent of which is methyl salicylate, which is formed from the glycoside gaultherin by the action of the enzyme gaultherase.

The fruit of a number of species of *Pygeum*, e.g., *P. gardneri* Hook. f., a middle-sized tree of W. India and the Nilgiris, and *P. acuminatum* Coleb., a moderate-sized to

FIG. 36 — *Agrimonia eupatoria* Linn.

large tree of E. Bengal and Khasi Hills, if crushed, emits a strong smell of bitter almonds. Similarly, the blaze (wood) of some species, such as *P. glaberrimum* Hook. f., a large tree of the E. Himalayas and Assam, *P. montanum* Hook. f., a small to medium-sized tree of the E. Himalayas and Assam, and *P. acuminatum*, also smells of bitter almonds. Even the leaves of some species smell of bitter almonds. Although none of the Indian species have so far been investigated, hydrocyanic acid has been reported from the bark of *P. parviflorum* Teijsm. & Binn. of Malaysia. The greenish-white or pale-yellow flowers of *P. montanum*, which are  $\frac{1}{2}$  in. across, are slightly scented.

*Eriobotrya bengalensis* Hook. f., a middle-sized to large tree of the E. Himalayas and Assam, bears white fragrant flowers which are  $\frac{1}{2}$  in. across.

*Potentilla fruticosa* Linn. (*pinjung*), a shrub in the temperate and subalpine Himalayas from Kashmir to Sikkim at altitudes of 8,000 to 12,000 ft., bears numerous, bright-yellow flowers 1 to 1 $\frac{1}{2}$  in. in diameter. Its pinnately compound fragrant leaves are said to be used as a substitute for tea by the local people, but no essential oil appears to have been distilled and examined. From the roots of some species of *Potentilla*, such as the W. Himalayan *P. anserina* Linn. and *P. reptans* Linn., a small quantity of an alcohol, tormentol, has been isolated.

*Agrimonia eupatoria* Linn., a herb of the Himalayas, Khasi and Mishmi Hills, is reported to contain an essential oil (Wehmer), the nature of which has not so far been investigated.

From among the constituents of essential oils reported from plants of this family may be mentioned: amyl alcohol, phenyl-ethyl alcohol, rhodinol (citronellol), geraniol, nerol, linalool, eugenol, farnesol, acetaldehyde, salicylaldehyde, nonylic aldehyde, benzaldehyde, furfural, citral, phenylacetic acid, methyl salicylate, coumarin and other lactones, amylamine, trimethylamine, cadinene, triacontane, and stearoptene.

Salicylic acid has been frequently identified in the distilled juices of several fruits, but it is not certain whether it pre-exists, as has been often claimed, in the form of methyl salicylate, in the juice of apple, strawberry, raspberry, cherry, and plum (Naves & Mazuyer, 1947. *Natural Perfume Materials*: p. 182).

## 1. CRATAEGUS Tourn. ex Linn.

(From the Greek *krataigos* — a flowering thorn.)

This genus comprises about 100 species which are usually spiny shrubs or small trees, the hawthorns, having white or pink flowers in terminal corymbs, and small berry-like pomes. In India it is represented by 4 species, all of which are confined to the Himalayas. Some species are cultivated in foreign countries on account of their ornamental flowers.

Although many species of this genus yield an essential oil, the hawthorn perfume of the market is not a natural product but is manufactured synthetically. The base for all perfumes of this type is anisaldehyde, but the odour is rounded off with acetophenone and finished with rose otto or other floral extracts.

Some of the constituents of the essential oils from flowers of some of the species are trimethylamine, amylamine, and sometimes ammonia. Similarly constituted oil has also been obtained from the leaves of some species, such as *C. monogyna* Jacq. of Europe.

### *Crataegus oxyacantha* Linn.

Fl. Brit. Ind., II, 383.

(Hawthorn, Maybloom, *Ban-sangli*)

This is a small tree with shining, often lobed leaves, white or pink fragrant flowers, and small red fruits often called "haws". It is found in the W. Himalayas from the Indus to the Ravi at altitudes of 4,000 to 9,000 ft. It is often cultivated for the sake of its flowers and edible fruit, which is said to be better than that of the European hawthorn.

The hawthorn has been regarded by the Greeks as a tree of fortune, and is said to be used for decorations during the May Day festivities of some European countries.

The flowers have a spicy odour, which recalls that of almonds. They yield 0.157 per cent. of an essential oil which contains trimethylamine (Wehmer). The natural oil is not an article of commerce.

## 2. GEUM Linn.

(The Latin name of herb bennet, *G. urbanum* Linn.; from the Greek *geuo* — to stimulate; referring to the aromatic roots.)

This is a genus of perennial herbs, having white, yellow or purple flowers. It

FIG. 37 — *Crataegus oxyacantha* Linn.

comprises about 40 species, which are natives of temperate and arctic regions, but only 2 are found in the temperate and alpine Himalayas.

***Geum urbanum* Linn.**

Fl. Brit. Ind., II, 342.

(Avens, Cloverroot, Herb Bennet)

This is an erect perennial herb with highly dissected leaves, yellow flowers  $\frac{1}{2}$  to  $\frac{3}{4}$  in. across, and hairy fruitlets. It is found in the temperate W. Himalayas from Muree to Kumaon at an altitude of 6,000 to 11,000 ft.

The root is aromatic, having a clove-like odour, and is sometimes chewed to sweeten the breath. Owing to its astringent

properties it has been employed in Europe in cases of dysentery and diarrhoea. It was also used to flavour ale in olden times.

Fresh comminuted roots, after standing for 12 hours, yield 0.1 per cent. of an oil on distillation, the bulk of which is eugenol (benzoyl eugenol). The roots contain a glycoside, "gein", which gets hydrolysed by an enzyme, "gease", also present in the root, leading to the formation of eugenol (Bourquelot & Hérissé, 1905. *C.R. Acad. Sci., Paris*, 140 : 870; Hérissé & Cheymol, 1925. *Ibid.*, 180 : 384).

The dry root was distilled by Haensel (1903. *Chem. Zbl.*, 1 : 1137), who obtained 0.022 per cent. of a reddish-brown oil with an aromatic odour, bitter taste, and sp. gr.  $13.5^{\circ}$  1.037.

FIG. 38 — *Geum urbanum* Linn.

### 3. PRUNUS (Tourn.) Linn.

(The classical name of the plum tree.)

This genus comprises 85 species of mostly unarmed shrubs or trees of north temperate regions. Of the 15 species found in India, the apricot (*P. armeniaca* Linn.), peach [*P. persica* (Linn.) Stokes], almond (*P. amygdalus* Batsch), sweet cherry or gean (*P. avium* Linn.), plums and prunes (*P. domestica* Linn.), *alucha* (*P. communis* Huds., var. *insititia* Hook. f.), cherries (*P. cerasus* Linn. and others) are well known.

The most important feature of this genus is the occurrence of cyanogenetic or hydro-

cyanic-acid-producing glycosides in the seeds of many of its representatives. These get decomposed by the enzymes (emulsin in almonds), which usually co-exist in the plant. Amygdalin, the glycoside of bitter almonds, is a typical example. It is also found in small amounts in peach kernels, plums, cherries, etc. In some plants the leaves also contain cyanogenetic glycosides, such as prulaurasin in the leaves of cherry laurel (*P. laurocerasus* Linn.), one of the ornamental shrubs of S. Europe.

*P. acuminata* (Wall.) Hook. f., a moderate-sized or small tree of the Central and Eastern Himalayas and Khasi Hills, bears small, white, scented flowers in axillary racemes.



# 1. *Prunus amygdalus* Batsch

*P. amygdalus* Baill., Fl. Brit. Ind., II, 313.

(Almond Tree, *Badam*)

It is a small to medium-sized tree which is cultivated in the cooler parts of the Punjab and Kashmir. It flowers profusely in the spring, before the leaves appear. The flowers are white, tinged with red. The drupes have a velvety, dry pericarp which separates into two valves when ripe. The stone is compressed, with shallow wrinkles and minute holes.

Two varieties of almond, viz., *amara* and *dulcis*, the bitter and the sweet, are well known even though no botanical characters are known to distinguish one from the other.

Apart from the fatty oil, the bitter almonds yield an essential oil, known as "oil of bitter almonds", to the extent of 0.5 to 0.8 per cent. The bitter almond oil containing hydrocyanic acid is a strongly refractive liquid which, when fresh, is colourless, but acquires a yellow colour on standing. It has the predominating smell of hydrocyanic acid which is present and should, therefore, be smelt with care. The oil has sp. gr. 1.045 to 1.070, acid val. 0 when fresh,  $[\alpha]$  0°,  $n$  1.532 to 1.544, and is soluble in 1 to 2 volumes of 70 per cent. alcohol. The content of hydrocyanic acid varies, being as high as 14 per cent. in the crude oil. It goes down on rectification.

Bitter almond oil from which hydrocyanic acid has been removed is a colourless, optically inactive liquid having b.p. 179°, sp. gr. 1.050 to 1.055,  $n$  1.542 to 1.546, and is soluble in 1 to 2 volumes or more of 70 per cent. alcohol. The above characteristics are comparable with those of benzaldehyde which is the main constituent of the oil that has been freed from hydrocyanic acid. Hydrocyanic acid is not present as such in the bitter almonds, but is formed during the hydrolysis of the glycoside amygdalin by the action of the enzyme emulsin which also is present in the bitter almonds.

In order to obtain the oil of bitter almonds, the fatty oil (which is present to the extent of 35 to 60 per cent.) is first extracted by pressing through a hydraulic press. The oil cake is then finely ground and suspended in 6 to 8 parts of boiling water and allowed to stand for a few hours. When it has cooled to about 50°C., about  $\frac{1}{2}$  part of fresh bitter almond paste in 6 to 8 parts

of water is added to start the enzymic action. The mixture is allowed to stand for 12 hours at about 50°C., by which time the fermentation should be complete. This mixture is now ready for distillation with steam. Hydrocyanic acid is highly poisonous and, therefore, fullest precautions should be taken to prevent its escaping into the air in a factory.

As has been stated before, the bitter and sweet varieties of almonds have no characteristic distinguishing features. Further, the sweet variety, which is far more common than the bitter, is devoid of the essential oil. For this reason and the high cost of almonds, the commercial oil of bitter almonds is mainly derived from the bitter kernels of some "varieties" of apricot (*P. armeniaca* Linn.) and peach [*P. persica* (Linn.) Stokes]. The essential oil of apricot and peach kernels is similar to that from bitter almonds. The yield of the essential oil of apricot kernels is 0.6 to 1 per cent. and that of the fatty oil 35 to 40 per cent.

The oil of bitter almonds contains benzaldehyde, hydrocyanic acid, and benzaldehydecyanhydrin (or mandelonitrile). The common adulterant of the oil is, therefore, synthetic benzaldehyde. Benzaldehyde gets readily oxidized to benzoic acid when left exposed to air. Bitter almond oil containing hydrocyanic acid, on the other hand, is much less prone to oxidation, the latter acting as a preservative.

Hydrocyanic acid can be removed from the oil of bitter almonds by mixing with water, red oxide of mercury, slaked lime, and ferrous chloride, out of contact with air, and heating on a water bath. The hydrocyanic acid gets removed and the oil rectified.

The United States Pharmacopoeia XII requires that the oil should not contain less than 80 per cent. of benzaldehyde, and not less than 2 per cent. and not more than 4 per cent. of hydrocyanic acid. This oil is intended for medicinal use and neither it nor its solution should be used for flavouring food. For flavouring purposes hydrocyanic acid is more or less completely removed, and such an oil is known as *oleum amygdalae*, S.A.P. (= *sine acido prussico*).

Even though the synthetic benzaldehyde is equivalent to the oil of bitter almonds, the latter is preferred by manufacturers of flavouring materials since it has a superior flavour due perhaps to small amounts of

FIG. 39 — *Prunus amygdalus* Batsch

other constituents, whose nature has not yet been investigated.

Bitter almond oil is used in pharmacy as a nerve sedative and as a base for soothing skin lotions. It is used in small quantities in such perfumes as heliotrope, and has also a wide application in the soap industry. It is also largely used as a flavouring agent in confectionery, liquors, etc.

## 2. *Prunus armeniaca* Linn.

Fl. Brit. Ind., II, 313.

(Apricot, *Khubani*, *Zardalu*)

The apricot is a medium-sized deciduous tree having pinkish flowers which turn white

after some time. The flowers appear before the leaves, and are either solitary or in bunches. The fruit is downy with a thickened and grooved margin. It is commonly cultivated in N.-W. India, especially in the hills at altitudes of 4,000 to 9,000 ft., and occasionally grows as self-sown.

Apricot is the commonest fruit in the Punjab Himalayas, and is eaten both fresh and dried. The seeds are eaten in the same way as almonds, and in fact form a valuable substitute. The oil expressed from these is largely used as an illuminant, for cooking, etc. As in the case of almonds, there is also a "bitter variety" of the seeds which is undoubtedly poisonous.

Apricot kernel of the bitter variety is the main source of the commercial "oil of bitter almonds". It yields 1.6 per cent. of the essential oil (Rabak, 1908. *U.S. Dept. Agr. Bur. Plant. Ind.*, 133: 23). The method of winning the oil is the same as described under bitter almonds. The uses of the oil are also similar.

### 3. *Prunus avium* Linn.

Fl. Brit. Ind., II, 313.

(Cherry, Sweet Cherry, Gean)

This is a medium-sized tree, with sweet or bitter, nearly black fruit having a smooth stone. It is cultivated in the N.-W. Himalayas up to an altitude of 8,000 ft., especially in Kashmir.

The fruit is well known and much valued as a dessert. The seed-kernel contains amygdalin (Wehmer).

### 4. *Prunus cerasus* Linn.

Fl. Brit. Ind., II, 313.

(Cherry, *Alubalu*, *Gilas*)

This is a deciduous shrub or a small tree bearing globose fruit which is light red or nearly black in colour, and acid or sweet in taste. The stone is smooth. It is cultivated in the Himalayas of the Punjab and N.-W. Frontier Province up to an altitude of 8,000 ft. The fruit is edible, and the kernel is used for flavouring liquors in Europe.

The seed-kernels as well as the leaves contain the cyanogenetic glycoside amygdalin (Wehmer).

### 5. *Prunus mahaleb* Linn.

Fl. Brit. Ind., II, 312.

(*Khewati*, *Mahaleb*)

This is a small, much-branched shrubby tree with white, fragrant flowers in peduncled corymbose racemes. The drupe is  $\frac{1}{4}$  in. in length; ovoid and tipped with a short, blunt point. It is a native of Central Asia and Central Europe, probably also occurring in N.-W. India. It is cultivated in Baluchistan.

The "scented kernels" are sold in the bazars of N.-W. India for use in indigenous

medicine (Watt). The seeds are stated to contain amygdalin (Wehmer), and, when chewed, have a strong odour of hydrocyanic acid (Dymock, Warden & Hooper, 1890. *Pharmacographia Indica*, 1: 567).

### 6. *Prunus padus* Linn.\*

Fl. Brit. Ind., II, 315.

(Bird Cherry, *Jamnoi*)

This is a small to medium-sized deciduous tree, the leaves turning red in autumn. The drupe is  $\frac{1}{8}$  in. in diameter, globose, acid to taste, of reddish colour turning to dark purple or black. It is a native of the temperate Himalayas, occurring at 4,000 to 12,000 ft.

According to earlier workers almost all parts of the plant (leaves, flowers, seeds, and bark) contain the cyanogenetic glycoside amygdalin (Wehmer). Recent work shows that the glycoside found in the twigs is prunasin (mandelonitrile glycoside) which, on hydrolysis, yields hydrocyanic acid, benzaldehyde, and glucose (Hérissey, 1912. *C.R. Acad. Sci., Paris*, 154: 1249).

The heartwood has an unpleasant smell when first exposed.

### 7. *Prunus persica* (Linn.) Stokes

*P. persica* Benth. & Hook. f., Fl. Brit. Ind., II, 313.

(Peach, Nectarine, *Aru*)

This is a large deciduous shrub or a small tree, bearing pink flowers which appear before or sometimes with the leaves. The fruit is succulent and its stone deeply and irregularly furrowed. Peach is a form with downy pericarp, and nectarine with a glabrous one.

Peach is widely cultivated in India, especially in the N.-W. Himalayas where some of the finest varieties of the fruit are grown. It also occurs in a naturalized state in the north-west up to an altitude of 10,000 ft. It is also cultivated extensively in the plains of the Punjab and N.-W. Frontier Province, and in the Nilgiris between altitudes of 5,000 to 7,000 ft.

The fruit pulp, on distillation and solvent extraction of the distillate, yields 0.0008 per cent. of a pale-yellow essential oil of peach odour. The reported constituents

\*Some authors prefer to call it *Prunus cornuata* Wall.

of the oil are acetaldehyde, furfural, cadinene and linalool, and esters of formic, acetic, valeric and caprylic acids (Power & Chesnut, 1921. *J. Amer. chem. Soc.*, **43**: 1725; 1922. *Ibid.*, **44**: 2966).

The seed-kernels contain amygdalin and yield about 0.7 per cent. of an essential oil, which contains chiefly hydrocyanic acid, benzaldehyde, and benzaldehydecyanhydrin (Rabak, 1908. *U.S. Dept. Agr. Bur. Plant. Ind.*, **133**: 23). These are one of the sources of commercial "oil of bitter almonds".

The flower, leaf, and bark have the peculiar odour and taste of bitter almonds, and are stated to contain hydrocyanic acid.

#### 8. *Prunus puddum* Roxb. ex. Wall.\*

Fl. Brit. Ind., II, 314.

(Wild Cherry of the Himalayas, *Padam*)

This is a moderate-sized to a large tree of brilliant appearance when in flower. The flowers are  $1\frac{1}{2}$  to  $1\frac{3}{4}$  in. in diameter, pink or white, and appear before the leaves. The drupe is  $\frac{1}{2}$  to  $\frac{3}{4}$  in. in length, oblong or ellipsoid, obtuse at both ends, with scanty, reddish or yellowish, acid flesh, and wrinkled and furrowed stone. It is found wild in the temperate Himalayas from Garhwal at altitudes of 3,000 to 6,000 ft. to Sikkim and Bhutan from 5,000 to 8,000 ft., and is also met with in the hill stations of Kodaikanal and Ootacamund in S. India.

The leaves and kernels are said to contain a substance which yields hydrocyanic acid (Greshoff, 1906. *Arch. Pharm., Berl.*, **244**: 670). The bark is also stated to contain a hydrocyanic-acid-yielding substance, and the smaller branches are used in indigenous medicine as a substitute for hydrocyanic acid (Watt).

The heartwood has an evanescent scent.

#### 9. *Prunus undulata* Buch.-Ham.

Fl. Brit. Ind., II, 316.

(*Aruwa*)

This is a medium-sized deciduous tree with rounded crown. It bears white flowers and ovoid drupes. It is found in the temperate Himalayas from Kumaon at altitudes of 6,000 to 8,000 ft. to Sikkim and Bhutan

from 8,000 to 12,000 ft., and also in the Khasi Hills.

The leaves and fruits contain substances which yield hydrocyanic acid (Greshoff, 1906. *Arch. Pharm., Berl.*, **244**: 670).

#### 4. *PYRUS* (Tourn.) Linn.

(The classical name of the pear tree.)

This genus comprises about 65 species of shrubs or trees of north temperate and cold regions. Economically it is an important genus in so far that fruits from many of its species, such as the apples (*P. malus* Linn.) and pears (*P. communis* Linn.), are edible and highly prized.

The essential oil is usually found in the fruit skin, though the flowers also in some cases have a smell. The flowers of *P. aucuparia* Gaertn., for example, have the smell of hawthorn (*Crataegus oxyacantha* Linn.) flowers, and the flowers of a foreign species are stated by Wehmer to smell of ammonia. Hydrocyanic acid is present in many species, particularly in their seeds.

#### 1. *Pyrus aucuparia* Gaertn.\*

Fl. Brit. Ind., II, 375.

(Mountain Ash, Rowan Tree, *Battal*)

This is a small tree with pinnate leaves, pink flowers  $\frac{1}{2}$  to  $\frac{1}{2}$  in. in diameter, and red globose fruits. It occurs in the temperate W. Himalayas from Kashmir to Kumaon at altitudes from 9,000 to 13,000 ft.

The flowers have the smell of hawthorn (*Crataegus oxyacantha* Linn.) flowers, and yield trimethylamine (Wehmer).

The leaves, buds, young twigs, and bark are said to contain amygdalin (Wehmer).

The fruit resembles in size and flavour that of the same tree in Europe. In certain parts of Europe it is made into a jelly, and also used in the preparation of a liqueur or cordial.

The defatted seeds were found to contain about 0.073 per cent. of hydrocyanic acid (van Itallie & Nieuwland, 1906. *Arch. Pharm., Berl.*, **244**: 164).

#### 2. *Pyrus malus* Linn.

Fl. Brit. Ind., II, 373.

(Apple Tree, *Seb*)

This is a small tree rarely exceeding 30 ft. in height. It is largely cultivated in

\*Some authors prefer to call it *Prunus cerasoides* D. Don.

\*Some authors prefer to call it *Sorbus aucuparia* Linn.

the Himalayas, the Punjab, Sind, N.-W. Frontier Province, Central India, and the Deccan. It is also found wild in the N.-W. Himalayas.

The apple is a well-known fruit, occupying the foremost place among fruits of temperate regions in importance and extent of cultivation. It also forms the basic raw material for the cider and pectin industries.

The fruit skin of apple yields, on distillation with steam, a very small quantity (0.00065 to 0.0035 per cent.) of an essential oil, which soon congeals. This on treatment with ether and subsequent treatment with alcohol can be separated into a solid part and an oily part. The yellow oily portion has the odour of apples.

The nature and the constituents of the essential oil differ with different horticultural varieties. The oil from the variety known as "Ben Davis", for example, contains acetaldehyde, amyl alcohol, furfural, formic, acetic, caproic and caprylic esters, a solid crystalline waxy substance of the nature of triacontane, etc. (Power & Chesnut, 1920. *J. Amer. chem. Soc.*, **42**: 1509). The oil from the "McIntosh" variety contains geraniol, citral, amyl ester, etc. (Wehmer). The proportion of these constituents varies according to the varieties, and this accounts for the differences in odour.

The seed-kernels contain amygdalin (Rosenthaler, 1912. *Arch. Pharm., Ber.*, **250**: 298).

## 5. ROSA Tourn. ex Linn.

(The classical name.)

This is a genus of well-known erect, sarmentose or climbing shrubs, the roses, having odd-pinnate leaves, mostly prickly stems, red, white, pink or yellow (never blue) flowers with many stamens, and urn-shaped calyx tube which becomes fleshy in fruit (rose hip) enclosing the achenes. It comprises about 150 species which are widely distributed in the north temperate regions and on tropical mountains. In India it is represented by eleven species (excluding those cultivated). Over four thousand horticultural varieties, with many petals, are recognized. These varieties, which have been mainly derived by repeated hybridization of about 30 wild species, are divided

according to habit or culture into 43 classes or races of which the following are the most important: climbing tea, polyantha, hybrid perpetual, hybrid tea, multiflora, Noisette, and tea. A number of species with numerous "varieties" have been introduced into India and are cultivated in public and private gardens, mainly for ornamental purposes. Not all of them are highly fragrant and rich in oil content; in fact some of them are more or less inodorous. It is only in a few that the oil content is high and they alone are fit for extraction of the oil. Of the indigenous species the following have fragrant flowers: *R. anserinaefolia* Boiss. of Waziristan and Baluchistan, *R. gigantea* Collett ex Crép. of Manipur, *R. macrophylla* Lindl. of the temperate Himalayas, *R. moschata* Herrm. (musk rose) of the W. Himalayas, *R. longicuspis* Bertol. of the Khasi Hills, and *R. sericea* Lindl. of the temperate Himalayas.

According to Parry, "It is of interest to note that the odorous organs of the rose may be divided into two categories—firstly, the petals of the flower; and secondly, certain green parts of the plant, which include the calyx and sometimes the green leaves. These two sets of organs work in the evolution of the perfume independently of each other, to such an extent that no case is known where the perfume of the two organs are identical in the same plant. For example, the *Rubiginosae*, of which the sweet briar is typical, has odourless flowers, whilst its leaves are delightfully fragrant. And when this rose is hybridised, as it has successfully been by Lord Penzance, the so-called Penzance briars (all hybrids) have sweet-scented foliage."

Only a few species of *Rosa* have been examined for their essential oil, and of these may be mentioned: *R. canina* Linn. (dog rose) of Europe and N. Asia, *R. damascena* Mill. (damask rose) cultivated in S. Europe, India, Arabia, Persia, Bulgaria, Turkey, and N. America, *R. centifolia* Linn. (cabbage rose, garden rose) cultivated in many countries including India, *R. gallica* Linn. (French rose) of middle and S. Europe and also grown in India, *R. moschata* Herrm. (musk rose) of N. Africa and India, *R. indica* Linn. of China and cultivated in India, and *R. alba* Linn. of Bulgaria but also grown in India. The characteristic rose perfume is well developed in *R. damascena*, *R. centifolia*, *R. gallica*, and *R. alba*. The species which, in Asia, is mostly grown for the preparation

of otto or attar\* is *R. damascena* with a very generous yield of flowers; *R. centifolia* is also used for this purpose.

The yield of the oil from different species is different, varying from 0.015 to 0.050 per cent. on fresh petals (or 1 part of oil in 2,000 to 6,000 parts of petals), and the nature of the constituents is also different. In general the constituents are geraniol, rhodinol (citronellol), linalool, eugenol, nerol, farnesol, phenylethyl alcohol, esters, citral, nonylic aldehyde, and stearoptene (Bertram & Gildemeister, 1894. *J. prakt. Chem.*, **49**(2): 185; 1896. *Ibid.*, **63**(2): 225; Schimmel & Co., 1890, Oct. *Rep.*; 1900, Oct. *Ibid.*: p. 53; Soden & Rojahn, 1900. *Ber. dtsh. chem. Ges.*, **33**: 1720; Soden & Treff, 1904. *Ibid.*, **37**: 1094).

Apart from the essential oil the rose flowers contain a bitter principle, red colouring matter, tannin (2.5 per cent.), fatty oil (1.7 to 2.6 per cent.), organic acids (about 3 per cent.), quercitrin, and a wax. The rose petal wax melts at about 60°C., and has been fully described by Prophète (1926. *C.R. Acad. Sci., Paris*, **182**: 1559). Along with the above about 2 per cent. of saccharose and 10 to 15 per cent. of invert sugars are also present. In India the rose petals are converted into a preserve with sugar and the preparation is known as *gulkand*. This preparation is used as a mild purgative and is a very pleasant medicine to take.

The rose hip is one of the richest sources of vitamin C, and for this reason was and still is in demand.

### 1. *Rosa centifolia* Linn.

Fl. Brit. Ind., II, 364.

(Cabbage Rose, Hundred-leaved Rose, *Gulab*)

This rose is often cultivated in Indian gardens. Next to *R. damascena* Mill., it is the most important rose used in perfumery. In India, however, it is seldom used for the extraction of the perfume. For further details, the reader is referred to what has been written under the account of *R. damascena*.

\*Although attar, according to various dictionaries, means otto or essential oil, in India it has come to mean, particularly in reference to rose, that oil which has been distilled in combination with some quantity of sandalwood oil.

### 2. *Rosa damascena* Mill.

Fl. Brit. Ind., II, 364.

(Damask Rose, Persian Rose, Rose, *Gulab*)

This is an erect shrub armed with curved unequal-sized prickles mixed with prickly and glandular bristles. The flowers are rosy pink, borne in bunches of 5 to 10. The sepals are bent backwards after flowering. Flowering occurs in February to April.

This is the commonest Indian garden rose, being cultivated for its beautiful fragrant flowers, and is the main species grown at Ghazipur, Hathras, Patna, Lahore, Amritsar, and several other places in India. This is the rose from which essential oil (otto or attar) and rose water have been manufactured in India for several centuries past.

The charm of the flower and its delightful fragrance have been appreciated since earliest antiquity, and for 5,000 years and more the rose has sweetened and often sanctified the life of man all the world over. From Persia and the Shah's own gardens one may trace its itinerary until, today, there is nowhere outside the frozen wastes where it does not bloom and give infinite joy. It graces the humble cottage and adds more than lustre to the vases in palaces. Rose throughout the ages has decked the bride and garlanded emperors and prelates. Indeed, no other flower or perfume has been so persistently popular as rose.

*History*: The distillation of roses probably originated in Persia even before the start of the Christian Era, but the earliest documentary evidence of rose water dates back to 810 A.D. At this period, in the reign of the Caliph Mamoun, the province of Farsistan was compelled to provide annually a tribute of 30,000 bottles of rose water to the treasury at Baghdad. Considerable quantities also used to be exported from Farsistan to India, China, Egypt, etc. The first authentic description of essential oil occurs in a work written by Geronimo Rossi of Ravenna who in 1574 observed small drops of the oil floating on the surface of rose water. In India the otto of roses is said to have been discovered by Nur Jehan, the wife of the Moghul Emperor Jehangir, in 1612 A.D., when she noticed a film of oily layer in a 'canal' filled with rose petals and water, in a palace in Delhi. She is said to have had this delicate perfume skimmed with a feather, and followed this practice to obtain her supply of rose oil. This discovery led

FIG. 40 — *Rosa damascena* Mill.

to the establishment of rose-oil and rose-water industry in India, particularly in Kanauj and Ghazipur where the industry has existed for at least 300 years, and even today Ghazipur and Hathras produce the bulk of whatever little rose oil or water is manufactured in India.

Rose cultivation in India is said to have been started about the seventeenth century, and in Bulgaria in the year 1710. By

1750 Bulgaria had become the principal source of supply of rose otto (Parry).

*Cultivation*: Rose plantations are raised almost invariably from cuttings. When cultivated for the purpose of oil, those varieties are selected which have a fine aroma, are rich in the yield of oil, produce larger number of flowers, and are resistant to climatic conditions. It is said that *R. damascena* satisfies these conditions,

even though in shape and appearance it is not so attractive as many other garden varieties. In point of fact, *R. damascena* is a cultural variety not known in wild state, and is presumably a hybrid between *R. gallica* Linn. and *R. canina* Linn. with the characteristics of the former predominating.

The cuttings from old bushes are made about the beginning of the cold weather (November) and planted in small pots or baskets. In about 3 months they root and by February leaves appear when they are removed from the pots, earth and all, and placed in beds of sandy loam, previously prepared, and manured with farmyard manure and superphosphate at the rate of one ounce to a square yard. The planting is done about 3 ft. apart and nearly double this distance between the rows, and in this way 2,500 plants can be accommodated in an acre. The plantation is watered freely and frequently during the hot and dry weather. Early and excessive drought is harmful to the rose crop and so is excessive rain that helps flourishing of several pests which may ruin the entire crop. By March the following year, i.e., 15 months after starting, the plantation should be ready for harvesting though the maximum amount of flowers is not to be expected till the fourth or fifth year.

In Bulgaria the plant is propagated through root cuttings. The old bushes are cut down and the roots pulled out and separated into as many as possible. These are then replanted four or five abreast, in long trenches about 18 in. wide and deep, manured and watered, so as to form one hedge 80 to 100 yds. long. The distance between two hedges is about 6 ft. In order to combat the fungal disease to which rose is liable, spraying with Bordeaux mixture (with 1 per cent. while the plant is dormant, with 0.5 per cent. just before the blossoms open, and again with 1 per cent. after flowering) is recommended (Georgieff, 1925 *Perfum. essent. Oil Rec.*, 16 : 441). Like in India, the planting is done in November, and by the following May young shoots appear and a year afterwards the plants are in full flowering. The maximum number of flowers appear between the fourth and tenth years. After about 10 years the plants are rejuvenated by cutting down the branches to ground level to allow new shoots to come up and form the original hedge. Rose shrubs thus handled produce flowers for 20 or 30 years.

It is well known that conditions of soil, irrigation, and climate are of prime importance in setting up this industry, since most extraordinary differences have been noted in the amount of otto yielded by the same rose when grown in different regions.

**Yield:** In Bulgaria one acre on an average yields 2,100 lbs. of flowers in one season (May) of 21 days (Gildemeister records 4,000 lbs. per acre in good plantations). A second flowering takes place sometimes in November, but, from the commercial point of view, it is said to be of little value. The yield of flowers in France varies from 1,800 to 4,500 lbs. per acre towards the fourth or fifth year, when the maximum production is realized. In Hathras, in the United Provinces, the yield of flowers is 500 to 600 lbs. per acre per year. Experiments on cultivation tried in Cawnpore, in the United Provinces (Srivastava & Sinha, 1921. *Perfum. essent. Oil Rec.*, 12 : 14), show that a yield of 1,200 lbs. may be readily obtained, and that there is a possibility of increasing this to at least 1,500 lbs. per acre per year.

**Collection:** Rose flowers are usually collected before they begin to open. The harvest lasts for 3 to 6 weeks and the crop must be worked up for the oil immediately. The gathering is done early in the morning before the sun gets hot. It is essential that the flowers be not picked with the hot sun on them because the flowers picked during the heat of the day not only give a lower yield of the otto but the otto also is not so sweet in fragrance. Not only are the yield and quality of the otto affected by picking the flowers during the hot part of the day but they are also affected by storage of the flowers.

Srivastava & Sinha (*loc. cit.*) have shown that the high temperature which prevails in Cawnpore, during April, has a marked influence on the yield of the otto. Experiments conducted right in the open under the heat of the sun and unprotected from the hot wind gave a yield of 0.01 per cent. of otto. Simultaneous experiments in a covered shed improved the yield to 0.015 per cent. When protected with wet screens to keep away the hot winds and to reduce the temperature of the air surrounding the receiver the yield improved to 0.02 per cent. Further, comparative experiments using freshly plucked flowers and flowers which had been kept for 24, 48 and 72 hours, respectively, showed that fresh flowers were the only ones suitable for distillation; those which had been kept



for 72 hours had lost the whole of the oil. Flowers picked before sunrise and distilled immediately gave 0.025 per cent. of otto. Flowers which are left for some time before distillation tend to get partially fermented though the fermentation can be prevented, to an extent, by salting and keeping in a cool place.

*Preparation of rose otto in Bulgaria:* According to Poucher (1936. *Perfume, Cosmetics & Soaps*, 2: 212), there are four systems of extracting the perfume from the flowers, and all of these are practised in Bulgaria. They are as under:

1. Open fire with small alembics.
2. Open fire with large alembics.
3. Steam stills including vacuum and rotary apparatus.
4. Volatile solvent extraction.

*"Distillation with Small Alembics"*—Up to 1902 all the rose otto exported from Bulgaria was distilled by the growers themselves in small tinned-copper alembics. At that time there were in all 2,800 distillers, operating over 13,000 small alembics having a total capacity of nearly fifteen million kilos of rose blossoms. Now there are only about 300 peasant distillers with under 2,000 stills, the old system of distillation being continually replaced by modern methods, involving the use of large improved stills. There are at present over 50 new modern plants, operating some 400 large open-fire stills and 30 steam stills. These modern factories distil nearly 80 per cent of the entire rose crop, and the growers only 20 per cent of it. The latter generally use the old-fashioned *gulapana*, which constitutes one of the out-buildings of the local farmer. The small alembics are some 40 to 43 inches high and have a base of about 32 inches diameter. There are two handles, one on each side for lifting when charging and discharging. They are constructed as near as possible to a stream so that the supply of water for distillation and condensation is assured. The apparatus is comparatively crude and consists of a truncated cone copper boiler having a capacity of about 120 litres. The helmet top is mushroom shaped and has a straight condensing tube fitted into one side. This runs through a wooden vat containing the cooling water, and where it emerges below there is a cavity in the ground which acts as a receptacle for the glass receiver. The stills are generally placed in rows on a low brick hearth. Wood is used as fuel.

"On arrival at the *gulapana* the red and white roses are mixed and distilled at once. During the busiest times it is often necessary to spread the roses out in a cool place to await distillation when they are moistened with cold water to prevent fermentation. Ten kilos of petals and 75 litres of water are placed in each boiler, the apparatus is fitted together and sealed. The fire is then lighted and it takes from 1½ to 2 hours for complete distillation. The yield of rose water and otto measures about 10 litres and this is placed aside. The apparatus is then emptied, the exhausted roses separated by strainer and the hot water returned to the boiler for the second distillation. This generally measures about 50 litres, and so another 25 litres of fresh water is added for the further charge of 10 kilos of rose blossoms. This process is repeated until the day's collection of flowers has been disposed off. The rose otto is separated from the reserved distillates by placing the products of four distillations (40 litres) together in the apparatus and carefully distilling the whole. The first 5 litres of cloudy distillate are reserved, and when this clears the otto is found floating on the surface. It is carefully removed and stored in glass flasks. The other 35 litres is left in the still and a fresh quantity of rose water added. The process is repeated and the otto separated.

"As will be observed, the peasant-farmer generally re-employs the residuary water remaining after each operation, and this constitutes a fundamental difference between his process and that of the larger manufacturers, where the exhausted flowers and *residuary water* are discarded. In the former case, the hot and dark coloured residuary waters give the farmer the advantage that he commences his new distillation with already heated water and so saves fuel. It will be obvious that the progressive concentration of extractive matter in these residuary waters will raise the boiling-point and cause constituents to be carried over with the distillate which increase the yield of oil. Since, however, such residuary waters in time assume a sharp and distinctly unpleasant odour, the increased yield of oil is largely discounted by the fact that the quality is impaired. Nowadays many farmers have observed this disadvantage and, like the larger distillers, completely discard both exhausted blossoms and water together, thus dispensing with the use of sieve or strainer as the case may be. The rose oil

made by this primitive method is known as *Peasant Quality* and possesses a soft, sweet, honeyed odour lacking in strength. It is cheaper than the oil distilled in the large alembics because, in the first place, the farmer does not 'cost' his labour and that of his family, and in the second place the capital involved is comparatively insignificant. This type of oil successfully competes with ottos produced by other means of distillation.

"*Distillation with Large Alembics* — There are over 50 large distilleries belonging to private firms or to co-operatives. These contain some 400 large open-fire stills having a capacity varying from 500 to 2,500 litres and are capable of distilling 100 to 500 kilos of roses at each operation. They are usually made of copper and may have either fixed or detachable heads. Different forms exist, some being the flat type of still and others the erect cylindrical apparatus. In all cases they differ from the small former type in that they are built into hearths so that no loss of heat occurs. Moreover, they have a perforated platform inside near the base to prevent the flowers coming into contact with the direct heat of the fires beneath. Worm condensers are almost invariably used, and the receivers resemble large florentine flasks having cylindrical glass tops for the observation of the oil as it collects on the surface. In the fixed-head type of still the flowers are charged through a manhole direct from the sacks and until the blossoms are within a foot of the top. Water is then run in until the flowers are completely covered.

"A hefty worker stirs the whole mass with a long pole to ensure the separation of the blossoms so that they will float freely once the operation has commenced. (Coagulated groups of flowers would interfere with the complete removal of the essence.) In normal times the ratio of water to flowers is strictly observed, being 4 to 5 litres to 1 kilo of roses. The ratio is only decreased with pressure of work when the crop is exceptionally abundant. The charging orifice is closed and the fire lighted. Considerable experience is here necessary. The application of heat must be gentle, otherwise a too sudden rise in temperature would drive out some of the lighter perfume constituents with the air remaining in the still and would thus be lost to the detriment of the subsequent oil distillate. As a rule it takes one and a half hours before any

condensation takes place, and from two to two and a half hours more to complete the distillation. The distillate passes through the condenser, which is comparatively hot at the top and not very cold at the bottom. The temperature of the distillate is kept at just over 35°C., otherwise the stearoptene would crystallise inside the condensing tubes. The distillation is not stopped until about 1 litre of rose water is collected for each kilo of flowers in the still, the actual volume depending upon pressure of work. The contents of the still, that is, exhausted flowers and water, are emptied from a side exit and pass down shoots into a river. The distillate flows from the condensers into the receivers, arranged in series of two for each still, and the rose water constantly passes from the second into open tanks having a capacity of 100 to 150 litres. When full, the contents are automatically pumped into large storage tanks for subsequent redistillation or cohobation. The oil floats to the surface of the receivers and appears in the glass cylindrical top already referred to. It is usually a pale yellowish-green crystalline mass, containing all the stearoptene and the more highly aromatic constituents. This is the **Direct Oil** and is known locally as *Surovo maslo*. It is removed by either pipette or spoon and transferred to glass bottles for the time being. The yield is comparatively small, because the major portion of the oil remains dissolved or is emulsified and in suspension in the rose water pumped into the storage tanks. This is known as the *first waters*, and when a sufficient quantity has accumulated it is transferred to the stills for cohobation or redistillation. About 1,200 litres are run into each still. The fire is lighted and heat applied gently. In an hour and a half the distillate begins to pass and the operation is completed in another hour. The condensers are operated cold since no stearoptene is present in this distillate. When some 150 litres of rose water have been collected, the distillation is stopped and roses added to the residual waters in the still. (The difference between this residual water and the peasant residuary waters above referred to will be apparent.) Distillation commences again for the production of direct oil. The above 150 litres of rose water constitute the *second waters* and are pumped into the storage tanks to join other first waters therein and to await cohobation. The oil which separates in the florentine flasks from the

distillation of the first waters is known as the **Water Oil** and is referred to locally as *Prevarka*. This is always fluid. It is removed and mixed with the **Direct Oil** to constitute the Rose Oil of commerce. The yield from the first and second distillation is, from the author's actual experience in Bulgaria, never a constant ratio; even though seemingly identical conditions were observed for repeated distillations. The approximate calculations made on the spot led to the estimate of 25 per cent direct oil as against 75 per cent water oil. These mixed runnings, as already stated, constitute the crude oil of roses. It is placed in glass bottles and exposed to the direct rays of the sun for a number of days, when impurities and colloidal matter are precipitated. The supernatant fluid is carefully decanted and filtered. Packing is done under the supervision of the local Excise authorities, who seal each of the well-known tinned copper vases and levy an export tax of about 4 per cent.

*"Steam Distillation"*—This is carried out by a few of the larger manufacturers who have very efficient and modern apparatus, often specially constructed after much expensive experimental work. The steam is usually generated in a building adjacent to the distillery and the stills are as a rule larger than those employed for direct fire, sometimes having a capacity of 3,000 litres. Two types are in existence, one having a steam coil or steam jacket and the other having a perforated direct steam coil. The latter have the advantage of more quickly raising the temperature of the water in the still and incidentally replacing part of that distilled over during the operation of the plant. For all steam stills the process, flower ratios, etc., are much the same as those already described. Owing to the ease with which the steam can be controlled it is obvious that this constitutes a distinct advantage over the direct fire systems. It is not unusual for some of the larger manufacturers to have both steam and direct-fire stills in operation together. The oils from the two sources differ in odour and the latter are usually somewhat stronger and sharper, even though the bouquet is less fine. From the author's comparisons on the spot, a mixture of the two would seem to make the ideal rose otto, blended in the ratio of 2 of fire to 1 of steam oil. The largest plant has over 70 fire stills, 2 enormous steam stills and 4 concrete batteries,

all of which were in continuous operation a few years ago during the peak period of perfumery sales. To-day, alas, most of them are idle.

*"Vacuum Distillation"*—There is one large plant in operation and the process differs slightly from the foregoing. In the first distillation no oil is separated, the product being entirely rose water which is pumped into storage tanks. It is then redistilled, and yields at one operation the whole of the oil. The odour of this rose otto is of a very special character and in the author's view would be exceptionally suitable for perfumes of the white rose type.

*"Rotating Apparatus"*—This a modification of the plant used in Grasse for the extraction of flowers with volatile solvents, and was introduced into Bulgaria by Charles Garnier many years ago. It consists of a fixed vertical drum, inside of which perforated metal drums containing the blossoms revolve on a horizontal axle. They continually dip into the boiling water at the bottom of the apparatus and the flowers are rapidly exhausted. The actual period of extraction is considerably reduced, roughly to half that required for the fixed apparatus. Moreover, the ratio of water to flowers is less, being somewhere between three to two and two to one. Furthermore, the quantity of rose water distilled over is only half of that necessary in the fixed apparatus. Whereas in the latter the ratio of water oil to direct oil is about three to one, it is about two to one in the rotating apparatus. The principal difference in the oil obtained is that of a much lower stearoptene with a consequent increase in the odour to weight ratio."

*Volatile solvent extraction*: According to Poucher, a few of the modern factories employ this process which was first introduced into Bulgaria by Garnier in 1903. He installed a factory at Kare Sarli, containing six 12-tray extractors (the *rove* type). Details of this method have been given by Poucher (*loc. cit.*) and also by Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 85, 235). According to Naves & Mazuyer six more factories were built after 1919, all but one equipped with stationary extractors; three of these were still working in 1939. Large quantities of flowers were treated in these factories, particularly from 1922 to 1930. In some years, the production of the concrete reached as high as

2,200 lbs., but fell to 450 to 650 lbs. between 1932 and 1935.

*Preparation of rose otto in Germany:* The Bulgarian peasant process is quite primitive and leaves much more room for improvement. In Germany roses picked in the morning are transferred directly to the large copper stills, each of which has a capacity of about  $1\frac{1}{2}$  tons of roses, in addition to the requisite water. The stills are not heated over direct fire but with steam; water is changed with every charge of flowers, and the otto is collected in a series of florentine flasks arranged as cascade. All this care results in producing a very superior otto and, although it has a high stearoptene content, it is twice as intensive in odour and it goes twice as far in perfumery blending. The yield of the otto is about 0.02 per cent.

*Preparation of rose otto in France:* The cultivation of rose is a very old industry in Grasse, the home of French essential oil industry. The rose which had till fairly recently been employed for distillation, almost to the exclusion of other varieties, was the well-known *R. centifolia* Linn.\* usually known as the May rose or *rose de Mai*. This rose is of exquisite fragrance, but requires much care in cultivation, and does not give a high yield of flowers. Its odour, however, is so charming that it is almost exclusively used for enfleurage purposes and for the preparation of rose water (Parry). Over and above *rose de Mai*, many new hybrids have been developed and they too are employed for the purpose of manufacturing concrete or otto. *Roses de Jardin*, which is a general name for all the garden roses like Brunner van Houtte and tea roses, have appeared in the market because of the higher yield of otto, even though their fragrance does not match that of *rose de Mai*. For instance, the fragrance of otto of van Houtte roses recalls that of a bouquet of mixed roses. This otto is distinctly green in colour when freshly distilled, but becomes yellow on keeping, melts at a lower temperature ( $15^{\circ}$  to  $16^{\circ}$ ), and has a high optical rotation frequently reaching  $-11^{\circ}$ .

\*According to several books on perfumery and essential oils, such as those by Finckmore, Parry, and Poucher the species cultivated mostly in France and known by the name of *rose de Mai* is *R. centifolia* Linn., but, according to the latest book by Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 234), it is *R. damascena* Mill. These authors also state that *rose de Mai* is the name for *R. damascena*.

According to Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 234), whereas the greater part of the Bulgarian harvest is used in the production of rose otto, in France only a small amount of the essential oil is produced, distillation being practised mainly to obtain the rose water. The major portion of the crop in France is used in the manufacture of concrete, but a small proportion is also used to produce pomades and oils by digestion. According to Poucher, approximately 70 per cent. of the flowers are treated by volatile solvents (principally petroleum ether), 20 per cent. by maceration, and the remainder by distillation.

*Preparation of rose otto in India:* Otto of rose and rose water have been prepared in India for the past several centuries, and the methods followed have been more or less similar in principle to those followed in Bulgaria. An account of a more recent attempt to establish this industry is given by Srivastava & Sinha (1921. *Perfum. essent. Oil Rec.*, 12: 14), and Gadre & Mukerji (1922. *J. Indian Industr.*, vide *J. Soc. chem. Ind., Lond.*, 1922, 41: 192R). Srivastava & Sinha (*loc. cit.*) state as follows:

"The two main centres of the rose industry in Upper India are Ghazipur and Hathras. Rose plantations exist in the neighbourhood of both these places, but the industry is confined to the manufacture of rose water and small quantities of attar (mixture of sandalwood oil and otto of rose).

"Very little, if any, pure otto is made at either of these places, the reason being that the yield of otto is so small as to make the manufacture quite unremunerative.

"Ghazipur was once renowned all over the world for its rose products, but now the industry has practically died out, and much of the so-called Ghazipur rose water really comes from Hathras, where roses can be grown more cheaply.

"(1) A series of experiments was carried out on the ordinary native still (called 'deg'), provided with an outlet pipe made of bamboo, and connected to a receiver, which is kept immersed in water. The experiments confirmed the results obtained last year, and a yield of 0.004 to 0.0045 per cent. of otto on the weight of fresh flowers was obtained.

"In all these experiments, freshly-plucked flowers, free from moisture, were used, and the conditions as regards quantity of flowers used, time of distillation, water used in the

still, were kept identical. The same distillation (*etc*) was used three times over, the quantity of flowers being the same in each case.

"(2) A second series of experiments was carried out, using the same apparatus as above and keeping all factors identical, with the improvement that the water used to cool the receiver was kept running. This brought up the yield to 0.005 per cent. This shows that the native practice of not changing the cooling water is responsible for some loss of the otto. Strange as it may appear, native distillers do not care to change the water, because they have an idea that cold water impairs the aroma of the otto.

"(3) An improved direct-fired still (cap. 20 gallons), devised and constructed by the Indian Essential Oils Company, provided with a worm condenser, was next tried. Several experiments were carried out, and the yield was found to have risen very greatly. All other conditions being the same, the yield was found to be 0.015 per cent. on the weight of the flowers.

"(4) Experiments carried out at Cawnpore on steam still using 10 lbs. steam pressure, gave a yield of 0.016 per cent."

Experiments on improved still (3 above) further showed that if the receiver was protected from hot winds and kept cool, using 100 lbs. of water and 25 lbs. of fresh flowers plucked before sunrise, the best yield was obtained, namely, 0.025 per cent. Hope is expressed that with a little further care the yield can be improved to even 0.03 per cent., which is the Bulgarian figure. Further, by improving the methods of cultivation it may be possible to increase the yield of flowers per acre as well as the oil content of the flowers (Srivastava & Sinha, *loc. cit.*).

The above experiments were repeated by Gadre and Mukherji (*loc. cit.*) and they state as under:

"The flowers used for distillation were those of *R. damascena*, the species employed in Bulgaria, and were of the red or pink variety exclusively, which is the commonest Indian garden rose. The flowers from Hathras were packed in tins with common salt and sent by rail to Cawnpore; they were distilled by the Indian Essential Oils Co., and the distillates were forwarded to the Government Technical Laboratory at Cawnpore for examination. The flowers grown around Cawnpore were distilled at the Government Technical Laboratory in a steam still and a direct-fired still which was

a modernised improvement of the Deg still. The flowers were collected early in the morning and distilled as soon as possible after collection. Distillation usually occupied from 3 to 5 hours and the weight of the distillate collected was the same as the weight of the flowers used. Double and triple distillations were also tried, the once distilled and double-distilled waters being mixed with more flowers and distilled from a copper still. In preparing these a layer of paraffin oil was placed in the receiver to minimise the escape of essential oil owing to the prevailing temperature and high winds. These layers of paraffin oil were afterwards collected and the amount of rose oil computed by determining the iodine-absorption figure of the paraffin oil. The oil content of the rose-water was similarly evaluated. Determined in this manner the average yield of otto obtained by distilling Cawnpore flowers amounted to 0.01 per cent. on the weight of the flowers.

"It was found that the iodine value determined by Wij's iodine solution gave satisfactory and concordant results. Calculations were based on 190 as the iodine-value for pure otto of rose, this figure being the mean values determined by Hudson-Cox and Simmons on various samples (Analyst, 1904, 29, 175) and agreeing with figures obtained by the authors for Bulgarian otto and a sample of pure otto made from Hathras roses.

"It was concluded from these investigations that rose flowers grown at various localities in the Aligarh District are superior for distillation to those available at Cawnpore or Ghazipur, the superiority being due to richer soil and better irrigation. By means of a preservative like common salt, the flowers can be stocked undamaged for 3 or 4 days after plucking. The high percentage yield of otto by the Indian Essential Oils Co. could not be substantiated by the authors; the average calculated yield of otto did not exceed 0.015 per cent., and the optimum yield was found to be much lower than that usual in Bulgaria and elsewhere. Determination of the iodine-absorption of rose-water in the manner described was found to be a ready and reliable method of evaluating the rose-water.

"The following types of stills were tried in the experiment:—The Deg type; the Bulgarian still; the steam-still in which steam is blown through the flowers; and an improved direct-fired still. All three stills gave practically the same results with a given variety of flowers. Live steam is

unsuited for making otto from rose-flowers. A steam-jacketed still would be ideal, but its use may not be practicable on rose fields. The fourth type of still was a modification of the Deg still with a separate top, containing a fairly wide leading tube which could be connected with a worm condenser. It is portable and easily worked."

In actual practice hardly any pure rose otto is manufactured in India. The so-called attar or "itr" of rose is a mixture obtained by the distillation of sandalwood oil and rose oil.

**Preparation of rose otto in other countries :** Otto of rose is also prepared in many other countries including Persia, Turkey, U.S.S.R., Spain, and N. Africa.

**Production of rose otto and its trade in important countries :** From 1927 to 1937 the area under rose cultivation in Bulgaria varied from 14,000 to 18,000 acres and the otto produced ranged from 4,000 lbs. to 7,000 lbs.

The total world production of rose oil during 1935-37 is said to be in the neighbourhood of 9,400 lbs. per annum.

According to Schimmel & Co. (1945. *Ann. Rep.* : p. 50), the area devoted to the cultivation of roses in Bulgaria decreased from 15,600 acres in 1939 to 5,700 acres in 1945. The yield of flowers was also below normal, because of neglect of plants, lack of fertilizers, and the planting of food crops among the rose plants. Only 770 lbs. of rose oil were produced in 1945 and no rose concrete was manufactured in that year or in 1944. Schimmel & Co. (*loc. cit.*), quoting from an article in the *Foreign Commerce Weekly*, 1946, 23(7), p. 41, give the following figures of production of rose oil and rose concrete in Bulgaria from 1938 to 1945 :—

	ROSE OIL lbs.	ROSE CONCRETE lbs.
1938	3,785	5,090
1939	8,310	190
1940	4,085	4,630
1941	1,840	2,650
1942	595	2,790
1943	2,490	4,435
1944	1,615	...
1945	770	...

In 1945 Bulgaria exported 1,408 lbs. of rose oil valued at \$753,300 to the U.S.A. and 284 lbs. to France. The U.S.A. did not import rose oil from Bulgaria in 1944, but France obtained 282 lbs. of the oil and 167 lbs. of rose concrete.

The crop of rose harvested in the Grasse area of France in 1945 amounted to only

588,000 lbs. as compared with 981,200 lbs. in 1944. Exports of rose oil from France in 1945 totalled 1,170 lbs. valued at Frs. 14,252,000.

Imports of rose oil from Morocco into France during 1945 totalled 1,980 lbs.

Imports of rose oil into the U.S.A. during 1945 included, in addition to the Bulgarian otto of rose (see above), 420 lbs. of rose oil from Turkey, 440 lbs. from the U.S.S.R. and 287 lbs. from France.

**Rose odours :** According to Parry, Blondel has grouped the rose odours as follows : rose, musk, mignonette, violets, lily of the valley, hyacinth, fruit, bugs, and clove.

According to Poucher, "Slight differentiations in odour value are not perceptible to every one. The sense of taste is, however, generally more developed, and inconsiderable modifications of flavour are more frequently noticed. To the trained specialist, however, the merest gradation of odour is appreciable, and an expert florist will name the variety of rose *even in the dark*. The real rose odour is unique, and represents a *type* which is undefinable, incomparable, and at present inimitable by synthesis. This type is best represented by *Rosa Damascena*, cultivated in Bulgaria, and approximated to very closely by *Rosa centifolia*, cultivated in Provence. There are many variations from this type, and some of the roses which exhibit slightly different floral notes are enumerated below ; they represent practically all those that the perfumer is required to imitate :—

"*R. arvensis*, also known as the Ayrshire rose — some varieties are myrrh-scented ; *Banksian*, recalling violets ; *Canina*, resembling mignonette ; *Desprez*, fruity ; *Eglantine*, whose leaves recall jasmin ; *Macartnean*, apricots ; *Marechal Niel*, the most delightful of the 'tea' class, having a somewhat fruity odour, resembling raspberry ; *Moschata*, growing wild in Tunis and believed by some to be musky and by others to resemble pinks ; *Muscosa*, moss-scented ; *May*, recalling cinnamon ; *Safrano*, recalling pinks ; *Socrates*, resembling the peach ; *Souveraine*, the melon ; *Unique jaune*, the most charming of the 'noisette' type and having an odour of hyacinths. During 1929 a new rose, known as 'Portadown Fragrance', made its appearance. It is alleged to have taken eleven years to 'produce' and is the result of ingenious crossing by Samuel McGreedy & Sons. According to report the odour is a complex of tea rose and verbena. In the

majority of cases red roses are more odorous than white ones, but a peculiarity possessed by a number of both kinds is that, when cut and placed in water, their fragrance appears more pronounced than when growing. Roses cultivated in a hot climate have a more powerful perfume, and it has been noticed that those flowers grown under glass develop a finer aroma than those which thrive in the open air. Some roses, *e.g.* *R. gallica* develop their perfume when dried, while others, *e.g.* *R. Damascena*, lose it under similar circumstances. Sawyer states<sup>1</sup> that before a storm the odour of a rose *seems* strangely increased, and suggests that this may be due to the oxidising influence of the ozone in the atmosphere or to the perceptive faculties being sharpened at such moments. Other peculiarities of the rose odour are (a) that no two flowers emit the same fragrance; (b) that different flowers from the same plant have never *exactly* the same perfume."

*Rose perfume*: The rose perfume is marketed in several forms, *e.g.*, otto or attar, concrete and absolute, rose water and oil of rose waters, and pomade. The best and most powerful form in which the perfume can be obtained is undoubtedly the otto. The otto, however, does not accurately represent the odour of the flower, since, during the process of distillation, a large proportion of phenylethyl alcohol is lost. Phenylethyl alcohol is a constituent of the rose oil and is highly soluble in water. During distillation of the otto it remains dissolved in water, and thus disappears to a very large extent from the otto itself. This fact accounts for the difference in odour, for example, between otto of rose and rose concrete or rose water. The odour of the rose absolute, which is prepared by using volatile solvents, is more nearly approximate to that of the flowers. The rose perfume may also be extracted in the form of pomade by maceration, and, in a few instances where rose water is prepared in Grasse, the oil is separated which contains the most highly odorous constituents and all the stearoptene. This is the costliest otto obtainable (Poucher).

One lb. of rose oil is obtained by distilling 3,500 lbs. of flowers in Bulgaria (Poucher).

*Characteristics of otto of rose*: The characteristics of otto of rose vary widely according to the locality in which the plants are cultivated, and also in one locality from season to

season, mode of distillation, and other conditions. All these factors have to be taken into consideration, and, therefore, the published data should not be regarded as final or complete. The odour of the otto also is subject to considerable variation. According to Poucher, there are two well-defined types, possessing: (1) the soft honeyed sweetness of the peasant oil, and (2) the intense sharpness of the essence (those distilled from the large fire stills). These two comprise the main part of the oils distilled, but in addition there are those which have (3) the aldehydic tone of the steam-distilled oils, and (4) the leafy heaviness of the product from vacuum stills.

Poucher states that in the first two types there are further variations depending upon (a) the ratio of different types of roses, (b) the time and care taken between plucking and distillation, (c) the particular locality from which the distiller obtains his flowers for the preparation of his own special bouquet, (d) the possibility in large fire stills of slight burning of the flowers, due to the contact with the walls, and (e) the type of the fire still, some preferring the detachable and others the fixed head. In order to obtain otto of reasonable uniformity, some of the larger distillers, therefore, prefer to blend oils obtained from different districts.

*Physical and chemical data for otto of rose*: The Bulgarian rose otto, generally speaking, is light yellow in colour, occasionally with a greenish tinge. It has the consistency of oil of almonds at 21° to 25°C. In taste it is pungent and balsamic and in odour strong. At about 18° to 21°C., acicular or lamellar crystals separate which are shiny and iridescent. On account of their low specific gravity they collect at the surface forming a thin film which breaks up readily when shaken. When cooled to a lower temperature the oil congeals to a transparent, soft mass which again liquefies with rise of temperature.

Higher stearoptene content tends to lower the specific gravity of the oil. On account of the difficultly soluble paraffins which it contains, oil of rose yields turbid solutions, even with very large amounts of 90 per cent. alcohol.

According to Simmons (1925. *Perfum. essent. Oil Rec.*, 16: 341), the following figures will be found to cover most oils of good quality; figures given by Poucher are also recorded for the purpose of comparison:—

1. "Odorographia" (1892), 24.



	SIMMONS	POUCHER
Specific gravity <sup>20°</sup>	0.850 to 0.860	0.849 to 0.865
Optical rotation	-1.3° to -4°	-1° to -5°
Refractive index <sup>20°</sup>	1.458 to 1.465	1.452 to 1.466
Melting point	19° to 24.5°	15° to 24°
Saponification value	7 to 18	...
Total alcohols, as geraniol	70 to 75%	63 to 84%
Citronellol	30 to 40%	24 to 64%
Stearoptene	14 to 20%	7 to 25%
Melting point of stearoptene	about 32°	...
Acid. val.	...	1.5 to 3.8
Ester val.	...	3.7 to 17.5

According to Simmons (*loc. cit.*), the most important constituents of the rose otto are geraniol (40 to 50 per cent.), rhodinol — citronellol (30 to 40 per cent.), nerol, phenylethyl alcohol, together with small amounts of esters, linalool, citral, nonylic aldehyde, eugenol, a sesquiterpene alcohol, and stearoptene.

Poucher records the following constituents in commercial samples of the rose otto: *l*-citronellol (24 to 64 per cent.), total alcohols as geraniol (63 to 84 per cent.), nerol (up to 10 per cent.), phenylethyl alcohol (up to 1 per cent.—more in oils by vacuum distillation), esters of geraniol (up to 3.5 per cent.), eugenol (up to 1 per cent.), stearoptene (7 to 25 per cent.), and traces of *l*-linalool, farnesol, citral, and nonylic aldehyde.

The characteristic honey odour has been attributed to traces of phenylacetic acid produced by oxidation of the phenylethyl alcohol. The proportion of these constituents varies in oils obtained in different countries. The presence of ethyl alcohol is reported in some cases, but it is ascribed to the decomposition of flowers, especially when the roses get heated and begin to ferment during transportation to the stills. If the petals were distilled as soon as they are picked, the oil contains no ethyl alcohol (Simmons, *loc. cit.*).

It must, however, be mentioned that the chemical analysis of an otto of rose can never be taken as a final criterion of purity, but should always be supplemented by a critical evaluation of its odour by an expert.

**Artificial otto of rose:** While no synthetic preparation has yet been able to displace the natural otto of rose, efforts to manufacture a real artificial otto have been unremitting. For this purpose combinations of the following have been employed: geraniol, linalool, the higher homologues of phenylethyl alcohol, citronellol, the various esters of these alcohols and of phenylethyl alcohol, phenylacetic acid and its esters, and substituted citronellols. A near approach to the desired rose smell is

obtained by suitable combination of these, but all such preparations lack in the freshness which comes only out of live flowers. In order to impart this freshness a little of the natural otto is always added to the artificial otto, and in good oils a generous proportion. Without the natural otto, the synthetic oil is of little value to the perfumers.

**Standards and examination of rose oil:** Rose oil with its high price has always been a profitable article for all adulterators. In this connection it may be mentioned that, in Bulgaria, the amount of otto exported in many years exceeded the amount actually produced. This is explained by the fact that adulteration was practised. In point of fact, at one time adulteration was the rule and pure otto of rose a rare exception. The universal adulterant employed in Bulgaria was the palmarosa oil. Pure geraniol and similar compounds are also used, and "artificial mixtures" are today available which are marketed solely for the purpose of encouraging adulteration. Phenylacetic acid, which is one of the constituents of the natural oil, is commonly used to impart to the adulterated product the honey odour, which is characteristic of the natural otto. Other adulterants employed are guaiacum wood oil, gurjun balsam, benzyl benzoates, nonylic and decylic aldehydes, etc. According to Schimmel & Co. (1938. *Ann. Rep.*: p. 91), adulteration by ethyl alcohol has been greatly on the increase during recent years, and that geraniol, geraniol fractions, and also citronellol are favourite adulterants. They also report a crude case of adulteration wherein copious amounts of easily detectable terpineol had been added.

Since 1935, the State Agricultural Bank of Bulgaria holds a monopoly in that country for superintending the manufacture and marketing of pure rose oil, and it may be hoped that it will be possible to obtain rose oil of pure quality. Nevertheless, rose oil is, and remains, an article that must be taken on trust, and ought only to be purchased from dealers of first-class reputation. With so costly a product as rose oil the danger of adulteration is very great.

As mentioned earlier the characteristics of rose oil vary a great deal, and it is difficult to draw up standard figures, even within very wide limits, to cover every genuine otto of rose. The bulking of oils from different districts, usually carried out



by the dealers in Bulgaria, eliminates most oils with very abnormal properties, and tends to something more approaching uniformity. Although no limits can be definitely laid down for pure otto of rose (even excluding French otto and those oils produced in steam vacuum stills), the characteristics of the commercial oils given earlier would be found to cover most oils of good quality. The effect on these figures of the various adulterants which are likely to be met with have been dealt with by Simmons (1925. *Perfum. essent. Oil Rec.*, 16: 341). He states as under:—

"SPECIFIC GRAVITY—The specific gravity of otto of rose is low, and is raised by practically all adulterants, except ethyl alcohol and added wax...

"OPTICAL ROTATION—This figure has been useful in the past in detecting the presence of gurjun balsam, which considerably raises the laevo-rotation, one sample examined by the writer showing as high a rotation as  $-19$  deg. The presence of the balsam can be readily confirmed by Dodge and Olcott's test, adding to 5 drops of the otto 10 cc. of glacial acetic acid and 3 drops of pure nitric acid, and well shaking, when with as little as 0.5 per cent. of gurjun balsam oil a reddish violet colour develops in about 30 secs. The introduction of gurjun balsam oil into otto appears to owe its origin to the adulteration of the otto with a rectified palmarosa oil, which had in turn been adulterated with gurjun balsam oil.

"REFRACTIVE INDEX—This figure, like the specific gravity, is raised by most adulterants except ethyl alcohol and added wax, which reduce it. Addition of ethyl alcohol is very easily detected by determining the refractive index before and after washing with slightly warm water, when an increase of .0015 after washing may be taken as equivalent to about 1 per cent. alcohol (vide 'P. & E.O.R.', 1915, p. 74). With genuine otto containing an appreciable amount of phenylethyl alcohol, the refractive index is very often slightly reduced by washing due to the extraction of this substance...

"MELTING POINT—The congealing point, which was formerly usually determined in the examination of an otto, seems now quite generally to have given place to the determination of the melting point. These points are usually defined respectively as the temperature at which the first crystal appears as the liquefied oil cools, and the

temperature at which the last crystal disappears as the solidified otto melts. For genuine oils the melting point rises with increasing proportion of stearoptene, but is reduced by the addition of geraniol, palmarosa, or geranium oils, and is raised by the presence of ethyl alcohol, since the melting point is really the temperature at which the other constituents, sometimes termed the 'eleoptene', dissolve the stearoptene, and the solubility of the wax is much reduced by the addition of alcohol. The solidifying point has been much more correctly termed by Siedler, the 'crystallisation point' (Schimmel, 'Semi-Annual Report', April, 1913, p. 90). The effect on the melting point of geraniol or similar adulterant is sometimes counterbalanced by the addition of stearoptene, spermaceti, or other wax. Provided the amount of stearoptene is not excessive, the presence of added stearoptene is difficult to detect, but the addition of spermaceti raises the saponification value of the otto, and is also readily revealed by the melting point and saponification value of the separated wax and the melting point of the fatty acids obtained after saponification. A sample of such wax separated from otto examined by the writer gave melting point  $49$  deg. C. and saponification value 118.7.

"SAPONIFICATION VALUE—The proportion of esters in genuine otto of rose is extremely small, and the determination of the saponification value therefore readily detects the presence of any saponifiable adulterant, such as spermaceti (vide supra), diethyl phthalate (found by Schimmel, 'Semi-Annual Report', April, 1913), or oils containing much ester, e.g., geranium oils.

"TOTAL ALCOHOLS—The determination of the total alcohols by acetylation serves to reveal the presence of added geraniol, palmarosa or similar adulterant, as with a genuine otto this figure rarely exceeds 75 per cent.

"CITRONELLOL—Umney has shown ('P. & E.O.R.', 1913, p. 328) that the determination of the percentage of citronellol by formylation gives distinctly useful information, this figure being depressed by adulteration with geraniol or palmarosa oil.

"STEAROPTENE—The significance of the proportion of this has been already pointed out. It is best determined by Burgess's process which consists in extracting 5 grms. otto with 25 cc. 85 per cent. alcohol, cooling

in ice for 5-6 hours, filtering off the stearoptene, washing once with cold 85 per cent. alcohol, and drying in vacuo over sulphuric acid for 12 hours.

"In addition to those mentioned above, other adulterants which have been noted in otto of rose include guaiac wood oil, and nonylic or decylic aldehyde (Parry, 'Chem. & Drug.', 1910, 531), though as nonylic aldehyde is a natural constituent of otto, adulteration with this substance might be difficult to prove.

"The chemical analysis of an otto of rose can never be taken as a final criterion of purity, but should always be supplemented by critical examination of its odour by an expert. How fallacious a chemical analysis only may be is well shown by the following figures, which were obtained for a mixture, prepared by the writer, of 85 parts genuine otto and 15 parts palmarosa oil, and all of which fall within the above-mentioned limits for a genuine otto of rose :

" Specific gravity at 30°C.	0.8554
Optical rotation ..	-1°30'
Refractive index at 25°C.	1.4592
Saponification value ..	11.2
Melting point ..	21.3°C."

According to Naves (1948. *Mfg. Chem.*, 19 (8): 371), for many years a rhodinol (citronellol) content of between 25 to 40 per cent. was accepted as a standard for a genuine oil, and oils with higher content were rejected as adulterated. Subsequent work has shown that the reverse was, in fact, true and that a low rhodinol content could be an indication of adulteration. Naves (*loc. cit.*) advocates a rhodinol content of not less than 34 per cent. as the criterion of a genuine Bulgarian rose oil. It has, however, been shown that the content of rhodinol varies between 23 to 40 per cent. in the products of the old type of apparatus, and between 45 to 55 per cent. by the modern apparatus; a value up to 63 per cent. might be obtained for the products of rotating apparatus.

Several authors have stressed the point that the ratio of rhodinol to geraniol falls during distillation. This accounts for the low figures for oil produced in "peasant" apparatus when the operation is pushed to the limit in order to obtain high yields at the sacrifice of quality of the essential oil. It is, therefore, correct to state that

the increase in the rhodinol content has been mainly due to improved distillation methods (Naves, *loc. cit.*).

According to Naves (*loc. cit.*), the products of rose are characterized by the presence of laevorotatory rhodinol, the rotatory power of which in sodium light is 4° to 4.50°. It is not possible to obtain such a product economically from another source. The rotatory power of rhodinol from geranium oil is never more than 2.5°, and the product from other sources is either racemic or dextrorotatory. It has, therefore, been recommended that rhodinol be isolated and its rotatory power checked by measurements of other constants, such as density, refractive index, etc. Isolation is effected as follows :—

The oil is heated at 140° to 160°C. in presence of an excess of benzoyl chloride, when all the alcoholic constituents are destroyed with the exception of the rhodinol and a part of the phenylethyl alcohol, which get converted into the benzoic esters. The low-boiling products are eliminated by distillation and the residue saponified. The alcohols are rectified by entrainment with steam, the phenylethyl alcohol is destroyed by heating at 140° over potash, and rhodinol isolated by fractional distillation (Naves & Glichitch, 1933. *Parfums de Fr.*, 11: 156).

Addition of rhodinol from any other source than rose oil depresses the rotatory power (Naves, *loc. cit.*).

#### Rose concrete

The rose concrete is extracted with petroleum ether or benzene. According to Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 234), the yield of petroleum ether concrete in Provence varies between 0.17 and 0.27 per cent. (usually between 0.24 to 0.265 per cent.). This concrete gives 55 to 65 per cent. of an absolute. They also state that in Bulgaria the yield of concrete by the same process is 0.22 to 0.25 per cent., and that this gives 50 to 60 per cent. of absolute. The concrete extracted with petroleum ether ranges in colour from light brown to dark brown, while that obtained with benzene varies from light green to greenish brown; the latter method gives a slightly higher yield. It is also stated that the roses cultivated at higher altitudes yield more concrete.

The petroleum ether concrete from May rose in Provence has the following characteristics: congealing point  $43^{\circ}$  to  $48^{\circ}$ , methoxy val. (Ziesel)  $49^{\circ}$  to  $54^{\circ}$ , acid val. 9.8 to 14.4, and ester val. 19.6 to 25.6. The Bulgarian concrete has the following characteristics: congealing point  $41^{\circ}$  to  $46.5^{\circ}$  and saponification val. 31 to 56. The commercial products are said to frequently contain a considerable amount of petroleum ether. The presence of petroleum ether depresses the congealing point (Naves & Mazuyer, *loc. cit.*).

The results of determinations of different samples, as given by Naves & Mazuyer (*loc. cit.*), show that absolutes from the Bulgarian rose have the following characteristics: sp. gr.<sup>15°</sup> 0.9682 to 0.9916,  $[\alpha]$   $+9.4^{\circ}$  to  $+14.3^{\circ}$ ,  $n_{20}^{\circ}$  1.50633 to 1.51556, acid val. 3.5 to 11.2, ester val. 19.6 to 30.9, and total alcohols 69.3 to 71.8. The absolutes are generally brownish green, and are limpid and syrupy. They have a remarkable dextrorotatory power, whereas the essential oils and the distillates are laevorotatory.

The distillates from the concretes and absolutes are colourless and limpid. The results of determinations of different samples, as given by Naves & Mazuyer (*loc. cit.*), show that the distillates from the Bulgarian rose have the following characteristics: sp. gr.<sup>15°</sup> 0.951 to 0.992,  $[\alpha]$   $-0.51^{\circ}$  to  $-2.4^{\circ}$ ,  $n_{20}^{\circ}$  1.5046 to 1.5190, acid val. 2.1 to 5.1, and ester val. 5.6 to 10.4 (Naves & Mazuyer, *loc. cit.*).

#### Rose water

In some countries, especially in India, the rose is distilled not for its otto but for the production of rose water. During distillation, the distilled perfumed water is not returned to the still, as is the case when the manufacture of otto is aimed at, but sold as rose water. Rose waters of different strengths are prepared. If, for example, 100 lbs. of rose petals are distilled to yield 100 lbs. of rose water, the first 50 lbs. distilling over is labelled "quadruple" and the next 50 lbs. "triple".

On the basis of his experiments performed in France, Poucher states that it requires 12,000 kilos of flowers to produce 9,000 litres of rose water, plus 1 kilo of direct oil.

The rose water can be evaluated "by repeatedly shaking with chloroform and

determining the amount of iodine absorbed by the chloroform extracts with Wij's solution, the time of interaction always being an hour, a period sufficient for complete absorption. Five successive extractions with chloroform were found completely to exhaust the rose-water of its oil. Once-distilled water made in the laboratory gave figures such as 0.0574, 0.0688, 0.0605, 0.0434 grams iodine absorbed by 450 c.c. rose-water, and thrice-distilled water gave 0.2156 gm. Thus a classification of samples of rose-water can be made by the iodine-absorption method" (Gadre & Mukerji, 1922. *J. Indian Industr.*, vide *J. Soc. chem. Industr., Lond.*, 1922, 41: 192R).

The districts of Ghazipur, Hathras, and Aligarh are the three main centres of the rose industry in Upper India, where considerable quantities of rose water are manufactured.

#### Oil of rose waters

In France, part of the large amount of rose water manufactured in Grasse is extracted with volatile solvents. The product obtained is the oil of rose water, which has different uses in perfumery than the essential oil. The oil of rose water can also be used for the reconstitution of rose water, and has the advantages of an economy of storage space, packing, transportation, and an assurance of better conservation. According to Naves & Mazuyer (*loc. cit.*), the yield by petroleum ether extraction is from 0.200 to 0.280 per cent. for the so-called "single" water manufactured at Grasse. This product consists mainly of phenylethyl alcohol.

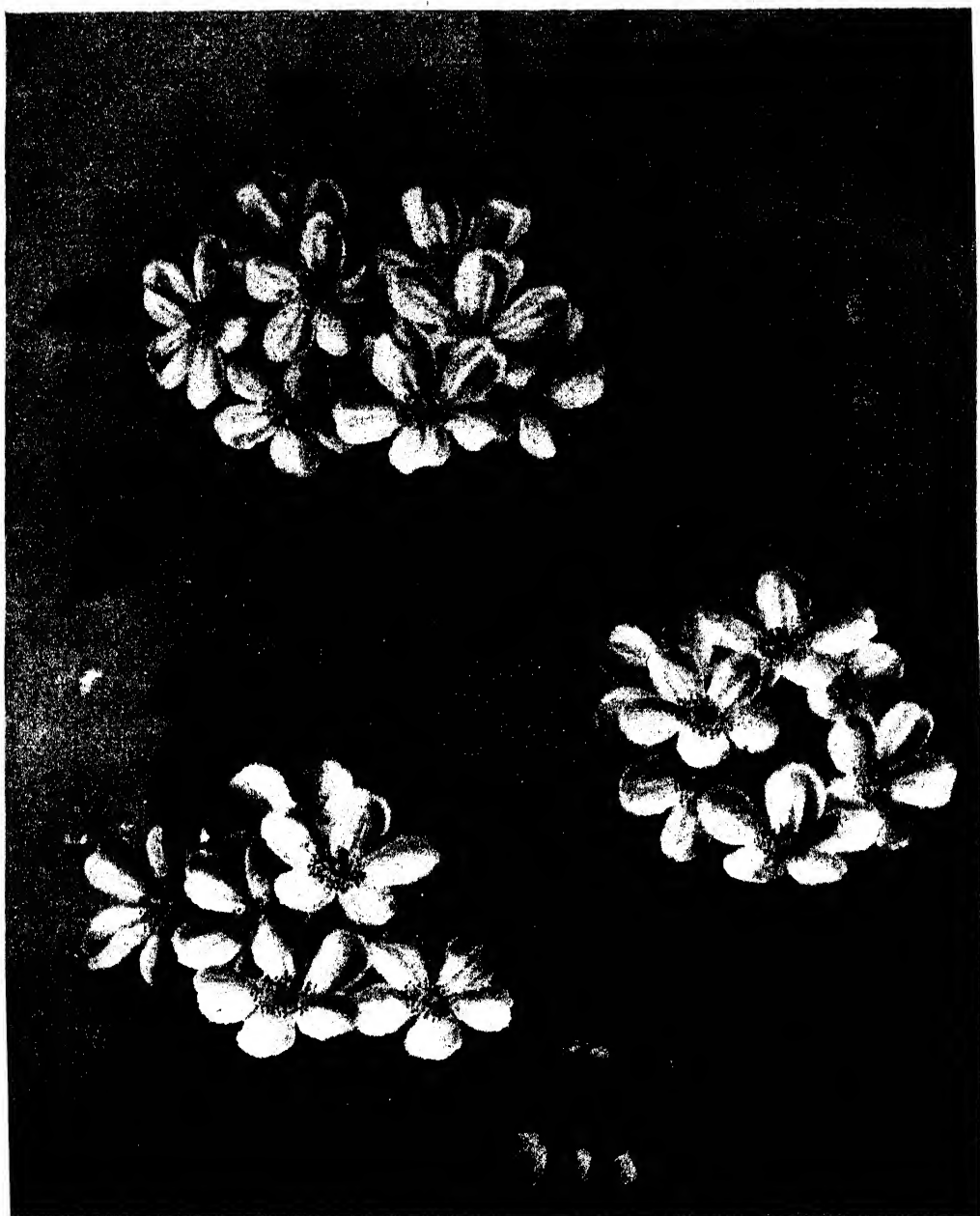
### 3. *Rosa moschata* Herrm.

*R. moschata* Mill., Fl. Brit. Ind., II, 367.

(Musk Rose, *Kuja*)

This is a large, thorny, climbing shrub with terminal compound corymbs of very sweet-scented, white flowers 1 to  $1\frac{1}{2}$  in. in diameter. It is found in the W. Himalayas at altitudes of 3,000 to 9,000 ft., but is more common between 4,000 to 6,000 ft.

According to Gupta (1928. *Kanjilal's Forest Flora of Chakrata, Dehra Dun, etc.*: p. 222), "An excellent otto is extracted from the flowers at Nahan in Sirmoor". The shrub flowers in May and June.

FIG. 41 — *Rosa moschata* Herrm.**6. RUBUS (Tourn.) Linn.**

(The Latin name for the bramble, derived from *ruber* — red ; referring to the colour of the fruit in some species.)

This is a large genus of often prickly shrubs, including the brambles, the black-

berry, raspberry, etc. Its members have 3- to 7-foliate or simple lobed leaves, white or pink flowers with a persistent calyx bearing the numerous stamens, and a mass of carpels ripening into a multiple fruit composed of many drupelets. This genus

is abundant in N. Hemisphere but rare in S. Hemisphere. Over 40 species grow wild in India, and some of them yield edible fruits.

The true raspberry of Europe is *R. idaeus* Linn. The fruit pulp, after extraction of the juice, yields on distillation a very small amount of a greenish oil of strong odour. The oil has sp. gr. 0.883,  $[\alpha] +2.8^\circ$ , saponification val. 193, the same after acetylation 215, and is soluble in 30 parts of 80 per cent. alcohol (Haensel, 1904. *Apothekerztg., Berl.*, 19: 854).

There are a number of species of *Rubus* growing in India which possess the odour and flavour of true raspberry, but it is not known if any of them yields an essential oil.

## 7. SPIRAEA Linn.\*

(From the Greek *speiraia*, from *speira*—a spire, something twisted; in allusion to the flexible branches.)

This is a large genus of unarmed shrubs or perennial herbs, with small perfect white

\* The genus as defined here is nowadays usually divided into a number of genera, the herbaceous members falling under *Ulmaria* and *Aruncus*, and the shrubby species under *Spiraea*, *Sorbaria*, etc.

or pink flowers in dense racemes, corymbs, cymes or panicles. Its members are found in the temperate and cold regions of N. Hemisphere, and about a dozen species are represented in India.

Members of this genus are hardly of any economic importance. Some of the Indian species, such as *S. bella* Sims, are ornamental and are cultivated in gardens. The Chinese *S. cantoniensis* Lour. is also occasionally cultivated. Both are shrubs; the former with pink, rarely white flowers, and the latter with white flowers.

The different parts of some species are stated to yield hydrocyanic acid. Thus, for example, the leaves, twigs and roots of *S. aruncus* Linn.\* of the temperate Western and Central Himalayas and flowers and leaves of *S. sorbifolia* Linn. of the temperate Western Himalayas are stated to yield hydrocyanic acid (Wehmer).

Salicylaldehyde has been reported from the leaves and flowers of some foreign species of this genus.

\* Some authors prefer to call it *Aruncus sylvester* Kostel.

*Supplement*

# AROMATIC PLANTS OF INDIA

## PART XI

BY

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### FAMILY

- 42. Saxifragaceae
- 43. Crassulaceae
- 44. Hamamelidaceae
- 45. Combretaceae



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# AROMATIC PLANTS OF INDIA

## Family XLII — SAXIFRAGACEAE

(Saxifrage Family)

THIS family comprises 90 genera and 750 cosmopolitan, chiefly temperate species of trees, shrubs or herbs. Its members are distinguished usually by the free bicarpellary ovary and by having as many or twice as many stamens as petals.

The family is of little importance from the economic point of view. Some species are cultivated in gardens, and among them may be mentioned *Hydrangea hortensia* Sieb. (garden hydrangea, Chinese guelder rose), *Philadelphus coronarius* Linn. (syringa), and species of *Saxifraga*, *Deutzia*, *Escallonia*, etc. Several species of *Ribes* yield edible fruits. The red currant (*Ribes rubrum* Linn.), black currant (*R. nigrum* Linn.), and the gooseberry (*R. grossularia* Linn.) are found wild in India and elsewhere, but are not cultivated in India for their fruits, as is done in some foreign countries.

The leaves of some species of *Ribes*, such as *R. nigrum*, *R. rubrum*, and *R. grossularia*, contain hydrocyanic acid. Tannins have been reported from the roots of some species of *Saxifraga*, saponins from the leaves of some species of *Saxifraga* and *Philadelphus*, and glycosides from some species of *Hydrangea* and *Dichroa*.

Essential oils have been isolated from the buds of *Ribes nigrum* and flowers of *Philadelphus coronarius*. The leaves and bark of *Itea rosmarinifolia* Poir. of Cochin-China contain an essential oil, but the Indian *I. macrophylla* Wall., *I. chinensis* Hook. & Arn., and *I. nutans* Royle have not been examined so far; the Indian species have glandular-toothed leaves. The rhizomes (*pakhan-bed*) of *Bergenia ligulata* (Wall.) Engl. (*Saxifraga ligulata* Wall.) of the Himalayas are said to contain an odorous principle (Dymock, Warden & Hooper, 1890. *Pharmacographia Indica*, 1 : 585); they are used in indigenous medicine as a tonic, in dysentery, and as a remedy for boils. The root bark of *basak* (*Dichroa febrifuga* Lour.) of the E. Himalayas is faintly aromatic (Dymock, Warden & Hooper, loc. cit. : p. 588), and is used in indigenous medicine as a febrifuge. *Deutzia corymbosa* R. Br., a shrub of the temperate

Himalayas, bears corymbose terminal panicles of white, fragrant flowers  $\frac{1}{2}$  to  $\frac{3}{4}$  in. in diameter. *D. staminea* R. Br., another shrub of the temperate W. Himalayas, bears panicles of white, fragrant flowers  $\frac{1}{3}$  to  $\frac{1}{2}$  in. in diameter.

*P-cymene* is the only constituent which has been identified in the black currant bud oil.

### 1. PHILADELPHUS (Riv.) Linn.

(The name of a plant mentioned by Aristotle, which has not been identified by modern botanists; from the Greek word *Philadelphos* — a sweet-flowering shrub.)

This genus comprises 20 species of shrubs of wide distribution in the north temperate regions. It is distinguished by the numerous stamens and inferior ovary. Several species with strongly fragrant flowers are cultivated as ornamental shrubs under the names of mock orange and syringa. In India it is represented by two species.

*P. tomentosus* Wall. (*P. coronarius* Linn., var. *tomentosus* C. B. Clarke), which is found in the temperate Himalayas, bears white, very fragrant, tetramerous flowers up to 1 in. in diameter.

### *Philadelphus coronarius* Linn.

Fl. Brit. Ind., II, 407 (without description).

(Mock Orange, Syringa\*)

This is an erect shrub with white, orange-scented flowers about 1 in. in diameter. According to the *Flora of British India*, this plant is not found in India, but Collet (1921. *Flora Simlensis* : p. 180) and some other authors report it from Narkunda and other areas in the Himalayas from Kishtwar to Sikkim at altitudes of 6,000 to 10,000 ft. It is also cultivated in some gardens in the hills. Parker (1924. *Forest Flora for the*

\* The common English name syringa should not be confused with the botanical name *Syringa*, which is a genus belonging to the family Oleaceae and includes the lilac *Syringa vulgaris* Linn.



FIG. 42 — *Ribes nigrum* Linn.

Punjab with Hazara and Delhi : p. 237 ), who calls it a European plant, states that this differs from *P. tomentosus* Wall. in having a septicidal ( not loculicidal ) capsule.

Treff, Ritter & Wittrisch ( 1926. *J. prakt. Chem.*, **113** : 355 ) extracted the flowers of various species of *Philadelphus* with petroleum ether. They reported 0.237 per cent. of a concrete and 0.124 per cent. ( 52.2 per cent. of the concrete ) of an absolute. The absolute on steam distillation yielded 0.006 per cent. of a yellowish distillate having a powerful and characteristic odour. The distillate had the following characteristics : sp. gr.  $^{15^{\circ}}$  0.947,  $[\alpha]$   $0^{\circ}$ , acid val. 28, ester val. 73, and ester val. after acetylation 224. It probably contained some methyl anthranilate. Farmiloe ( 1929. *Perfum. essent.*

*Oil Rec.*, **20** : 321 ), who investigated the flowers of *P. coronarius*, obtained 0.25 per cent. of a petroleum ether concrete, which yielded 38.2 per cent. of an absolute. The odour of the absolute approximated to that of jonquil and narcissus.

Igolen ( 1938. *Parfums de Fr.*, **16** : 92 ) recovered 0.144 to 0.179 per cent. of a reddish-brown petroleum ether concrete of hard consistency. This concrete gave 25 to 27.2 per cent. of a reddish-brown absolute of thick consistency, having a penetrating, fruity odour suggestive of the syringa flowers. From the absolute, Igolen obtained 9 per cent. of a steam distillate, which had the following characteristics : sp. gr.  $n^{15^{\circ}}$  0.912,  $[\alpha]$   $+3.5^{\circ}$ ,  $n^{20^{\circ}}$  1.4668, acid val. 25.2, and ester val. 95.2.

The natural perfume is not extracted, or, if so, only in insignificant quantities. The synthetic perfume of this name is usually composed of terpineol, linalool, hydroxycitronellal, and methyl anthranilate.

## 2. RIBES Linn.

(From the Arabic name *ribas*, meaning a plant with an acid juice.)

This genus comprises about 60 species of shrubs out of which 8 are represented in India. Its representatives have small, racemose, variously coloured flowers with 4 or 5 scale-like petals, 4 or 5 stamens, 2 styles, and an inferior ovary. The fruit is a globose or ovoid berry. It includes the currants and the gooseberry which have been referred to in the account of the family.

Hydrocyanic acid and essential oils have been reported from some species.

Several Indian species are covered with resinous glands or glandular hairs. For example, the twigs of *R. orientale* Desf. of the W. Himalayas are glandular and sticky, and its pubescent leaves have coarse sticky-glandular hairs. *R. nigrum* Linn. has been investigated for its essential oil, and is dealt with below.

## *Ribes nigrum* Linn.

Fl. Brit. Ind., II, 411.

(Black Currant, *Muradh*)

This is an erect aromatic shrub 6 ft. high. The young twigs are hairy and clothed with sessile yellow resinous glands which are also found on the petioles, lower surface of the leaves, inflorescence, and calyx. The heart-shaped 3- to 5-lobed leaves are scented. The flowers are greenish, on 2 to 3 in. long racemes which arise from leaf-producing buds. The berries are globose,  $\frac{1}{2}$  in. in diameter, black, with a strong aromatic smell and taste. It is found in the W. Himalayas, from Kunawar to Kashmir, at altitudes of 7,000 to 12,000 ft.

The plant is often cultivated in Europe for its edible fruits. The Indian fruits from wild plants are said to be similar to the cultivated black currants.

The buds yield 0.75 per cent. of a pale, greenish oil, having the odour of cymene. Its characteristics are: sp. gr.<sup>15°</sup> 0.8741,  $n_D^{20}$  1.4858,  $[\alpha]_D^{20} +2.3^\circ$ , and ester val. 5.6. The oil is soluble in 6.5 parts of 90 per cent. alcohol, with slight cloudiness, but is clear in 10 volumes (Schimmel & Co., 1907, Apr. Rep.: p. 106).

The leaves are said to contain an essential oil, quinic acid, and a very active oxydase (Huchard, 1909. *Pharm. J.*, 82: 528).

## Family XLIII — CRASSULACEAE

(Orpine and Houseleek Family)

This is a small family of succulent herbs or undershrubs, with free calyx, persistent petals, and fruit consisting of distinct carpels. It comprises 25 genera and 450 cosmopolitan species found chiefly in S. Africa.

A large number of plants of this family have smooth succulent leaves, and are able to grow in places where water is at times scarce. Life plant [*Bryophyllum pinnatum* (Lam.) Kurz, syn. *B. calycinum* Salisb.] is remarkable for forming new plants at the crenatures of the leaves when these are laid on damp ground. A few species are cultivated for ornamental purposes, for example, *Rochea coccinea* DC. of the Cape of Good Hope. Some species of *Cotyledon*, especially

*C. ventricosa* Burm. f. and *C. wallichii* Harv. of S. Africa, are said to be concerned in the induction of a disease called "krimpsiekte" or cerebro-spinal meningitis, especially in small stock.

The only species reported to contain an essential oil is *Sedum telephium* Linn. (orpine) of middle Europe. Its leaves, stems, and roots contain an amorphous glycoside, which breaks up through the agency of emulsin into glucose and geraniol-smelling essential oil (Bridel, 1922. *J. Pharm. Chim., Paris*, 26 (7): 289; 1922. *C.R. Acad. Sci., Paris*, 174: 186; 1923. *Ber. Schimmel u. Co. Lpz.*: p. 213).

## Family XLIV — HAMAMELIDACEAE

(Witch-hazel Family)

This family comprises about 20 genera and 50 species of shrubs and trees which are chiefly found in the subtropical region.

The family, though a small one, is important from the economic point of view. Some species, such as those belonging to the genera *Altingia*, *Bucklandia*, and *Liquidambar*, yield valuable timbers. Others, such as those belonging to the genera *Hamamelis*, *Corylopsis*, and *Distylium*, are grown as ornamental shrubs. The witch-hazel or hamamelis extract of trade, which is used in medicine as an astringent and to stop bleeding, is obtained from the leaves of witch hazel (*Hamamelis virginiana* Linn.) of N. America. The tough and flexible twigs of *Parrotia jacquemontiana* Decne. of N.-W. Himalayas are largely used in wickerwork in the same way as the willows, and also twisted into thick ropes, often 300 ft. long, which serve for crude suspension bridges across the hilly streams. The wood of this species is esteemed for walking sticks, tent pegs, rice pestles, etc.

The balsam storax or styrax, which is obtained from some foreign species of *Liquidambar*, is of great importance in perfumery, and is also valued in medicine as a parasiticide in scabies and other parasitic skin affections. The Indian *Altingia excelsa* Noronha also yields a balsam, rasamala, which, though differing from the genuine storax in some respects, may be used as a substitute.

The leaves and branches of *Hamamelis virginiana* on steam distillation yield a soft, sticky, green oil of strong odour. When this is distilled with steam, an yellowish oil of a powerful odour is obtained. The bulk of this oil consists of a sesquiterpene, an alcohol (7 per cent.), a small quantity of ester, and a wax (about 70 per cent.).

Among the constituents of essential oils isolated from members of the family may be mentioned:  $\beta$ -phellandrene and other sesquiterpenes, dipentene,  $\alpha$ - and  $\beta$ -pinene, camphene, borneol, styrene (styrol, phenylethylene), cinnamic alcohol, cinnamic acid, cinnamyl cinnamate, coumarin, vanillin, benzaldehyde, cinnamaldehyde, and unidentified aldehydes and ketones.

### 1. *ALTINGIA* Noronha

This genus comprises three arboreal species which are found in Assam, S. China,

and southwards. In India it is represented by only one species, *A. excelsa* Noronha, which is the source of a product called Burmese storax. Tonkin storax is derived from *A. gracilipes* Hemsl.

### *Altingia excelsa* Noronha

Fl. Brit. Ind., II, 429.

(Rasamala, *Jutuli*)

This is a tall aromatic tree found in the forests of Assam and Bhutan. It is also found in Tonkin and thence southwards through Burma to Sumatra and W. Java.

The wood is extensively used by planters in Assam for building purposes. The heartwood has a sour odour when first exposed, but this passes off as the timber seasons.

The tree yields a resin which is called rasamala. The name rasamala has been applied not only to the product from this tree, but also to the Oriental storax from *Liquidambar orientalis* Mill. from Asia Minor and several other balsams with a similar odour.

The resin does not appear to be collected in Assam. According to Burkill (1935. *Dictionary of the Economic Products of the Malay Peninsula*, 1, 118), "In the Levant, trees of *Liquidambar orientale* (sic) are exploited for liquid storax, first by stripping off the outer bark, then by scraping off the inner bark, which is packed in horse-hair bags and pressed, soaked in hot water, and pressed again, the resin being extracted by the two pressings; or the bark is boiled and then pressed. The bark is afterwards dried and used for fumigations. In the East there is no such process, but the bark is slashed, and the scanty clear resin collected; or fire may be applied to a hole cut in the tree and discoloured resin obtained. Damage to the tree is, in any case, necessary before the resin is got. The resin which runs from clean wounds is in the form of clear tears, which adhere but slightly to the bark. It exudes very slowly. No resin, apparently, is collected in Assam; but there was a collecting-industry in Burma, and there is one still in Annam. It may be collected in Sumatra and Java, but scarcely comes into the market where what is sold as rasamala is generally the imported storax of *Liquidambar orientalis*.

FIG. 43 — *Allingia excelsa* Noronha

The resin of *A. gracilipes* is collected similarly in Tonkin."

The resin differs considerably in its composition, according to the locality and the manner in which it is collected. Details of the different kinds of resins obtained from this species are given by Tschirch & Stock (1935. *Die Harze*, 2(1) : 105).

According to Hooper (1904. *Agric. Ledger* : p. 118), Manson slashed rasamala trees in Burma and obtained a white honey-like exudation from some of the wounded trees, but not from all of them. He noticed that the crust of resin near old wounds was hard and black. Manson also employed the method of obtaining resin from dipterocarps

(by the application of fire?), and recovered a dark-brown solid resin.

Schimmel & Co. (1892, April. *Rep.* : p. 56) obtained 0.17 per cent. of an essential oil by distilling the wood of Dutch East Indies origin. At ordinary temperature this oil formed a semi-solid crystalline mass having an odour resembling that of cinnamon and rhubarb. The principal constituent of the oil was a crystalline substance which melted at 54° to 55°C., and probably a ketone of which the oxime melted at 160° to 107°C. According to Gildemeister & Hoffmann, however, it is not certain whether the wood subjected to distillation by Schimmel & Co. was from *A. excelsa*

or any other tree known by the name of rasamala.

Tschirch & van Itallie (1901. *Arch. Pharm., Berl.*, 239 : 541) have reported cinnamic acid, benzaldehyde, and cinnamaldehyde from the resin obtained from Java.

Hooper (*loc. cit.*) examined two kinds of balsams from Burma. The one which resembled honey in colour and consistency when fresh became white and crystalline after two years and possessed a delightful fragrance of styrene. This "soft white crystalline balsam" contained 7.65 per cent. of a volatile oil. The total cinnamic acid content in the resin was 37 per cent. The resin had the following characteristics: acid val. 24.96, ester val. 174.39, saponification val. 199.35, and iodine val. 57.3. The other sample consisted of a dark-brown solid balsam with a strong and pleasant odour, that of cinnamon predominating. It contained 38 to 41 per cent. impurities, traces of free cinnamic acid, and 9.7 per cent. combined cinnamic acid. The pure resins amounted to about 54 per cent. and the essential oil to about 6 per cent. The characteristics of the balsam after purification with alcohol were: acid val. 76.8, ester val. 53.64, saponification val. 130.44, and iodine val. 51.68.

Hooper (*loc. cit.*) confirms Tschirch & van Itallie (*loc. cit.*) that the Burmese resin, like that of Java, has a different constitution from true storax of Asia Minor. At the same time he states that the resin is rich in aromatic compounds.

The resin from Assam trees has been recently investigated at the Forest Research Institute, Dehra Dun. It is a pale-yellow odorous resin and is obtained in clear tears from clean wounds. It contains 4.4 per cent. of essential oil. The resin extracted by 95 per cent. alcohol has m.p. 90° to 95°, sp. gr.<sup>20</sup> 1.046, acid val. 58.3, saponification val. 105.4, and ester val. 47.1 (Unpublished Records of the Forest Research Institute, Dehra Dun).

## 2. LIQUIDAMBAR Linn.

(Means *liquid amber*, referring to the fragrant resin which its species yield.)

This genus comprises four arboreal species which are found in the Mediterranean region, Asia, and N. America. All the species yield fragrant balsams, known in commerce as storax or styrax, but that from *L. orientalis* Mill. of Asia Minor is the most important, and called Oriental or Levant storax. This

is a semi-liquid, greyish-brown, sticky, opaque substance with a pronounced aromatic odour. Next in importance is the American storax which is obtained from *L. styraciflua* Linn. (sweet gum). This balsam is a thick, clear, brownish-yellow, semi-solid or solid substance. The Chinese storax is obtained from *L. formosana* Hance.

Storax is highly prized by the perfumer on account of its fixative properties and its sweet, heavy, "Oriental" type of odour. The purified balsam is largely used in soap perfumery and cosmetics, and in perfumes of the type of hyacinth, narcissus, hawthorn, cassie, magnolia, etc. It is also used in incense, as a flavouring for tobacco, and as an important ingredient of many pharmaceutical preparations.

In Asia Minor, the outer bark of the tree is bruised and beaten in order to promote the secretion of the balsam. The outer bark is then removed and sold for fumigation purposes. The inner bark is then stripped off and boiled in water, when the balsam floats on the surface whence it is removed. The bark is also expressed subsequently (Poucher). For another and slightly different account of the collection of storax the reader is referred to an article by Jeancard (1925. *Parfum. mod.*, 18 : 73). The crude balsam, known as liquid storax of commerce, is further purified by dissolving in alcohol, filtering to remove portions of bark and other debris, and evaporating to remove the solvent; it is then called "prepared storax".

According to Finemore, the Oriental balsam contains free cinnamic acid (23 per cent.), combined cinnamic acid (24 per cent.), aromatic esters (22.5 per cent.), styrene and vanillin (2 per cent.), resin (36 per cent.), water (14 per cent.), insolubles in ether (2.4 per cent.). According to Naves & Mazuyer (1947. *Natural Perfume Materials*: p. 265) a good quality balsam gives 60 to 75 per cent. of an alcoholic resinoid, 55 to 70 per cent. of a benzene resinoid, 35 to 55 per cent. of a petroleum ether resinoid, and 65 to 70 per cent. of an acetone resinoid. These resinoids are prepared with heat. Storax is known to contain a high percentage of water; sometimes the water may reach 20 or even 30 per cent. Storax purified with alcohol has the following characteristics: acid val. 68 to 94, ester val. 105 to 154, and saponification val. 171 to 234.

The balsam yields 0.5 to 1 per cent. of a highly aromatic oil (storax oil). This oil



FIG. 44 — *Terminalia arjuna* Wight & Arn.

is a bright-yellow to dark-brown liquid of varying constitution, which depends upon the manner and style of distillation. It contains styrene (styrol, phenylethylene), which is the principal odorous constituent having an unpleasant, sharp, benzene-

like smell, cinnamaldehyde (styrone), with the ethyl, benzyl phenylpropyl esters of cinnamic acid, and a trace of vanillin. Some cinnamyl cinnamate distils over with it. Naphthalene has also been isolated from the oil (Finnemore).

## Family XLV — COMBRETACEAE

( Myrobalan Family )

This family comprises 16 genera and about 500 species of trees and shrubs, some of the latter climbing, with alternate or opposite, simple, entire leaves and perfect or rarely polygamous flowers. Its representatives are widely distributed throughout the tropical and subtropical regions of the world.

From the economic point of view this is an important family, containing as it does some of the important and widely distributed trees of India, which are valuable for their timber and other products. Several species of *Terminalia* and *Anogeissus* are especially important for their timber. The fruit and bark of many plants, especially the terminalias, have astringent properties due to the presence of large quantities of tannins; they are often used medicinally. The fruit of *Terminalia chebula* Retz. (chebolic myrobalan, *harar*) is a valuable tanning material, and large quantities of it are exported, valued at about Rs. 4,500,000 annually. That of *T. bellirica* (Gaertn.) Roxb. ex Fleming (belleric myrobalan, *bahera*) enjoys considerable internal trade for use in indigenous medicine, as a purgative, etc. The gum (ghatti gum) from *Anogeissus latifolia* Wall. is commercially important, being a good substitute for gum tragacanth.

*Quisqualis indica* Linn. (Rangoon creeper) is commonly cultivated in Indian gardens.

Members of this family have not been investigated from the point of view of essential oils, but a number of them bear scented flowers. The small flowers of *Terminalia alata* Heyne ex Roth, syn. *T. tomentosa* Wight & Arn. and *T. tomentosa* Bedd., var. *typica* C. B. Clarke (*laurel*, *ain*, *saj*), *T. arjuna* Wight & Arn. (*arjun*), *T. catappa* Linn. (Indian almond tree, *jangli badam*), *T. travancorensis* Wight & Arn., syn. *T. angustifolia* Roxb. (*kotta-kadakai*) are sweetly scented. The flowers of *T. chebula* Retz. and *T. bellirica* (Gaertn.) Roxb. ex Fleming have an offensive odour. All these are medium-sized to large trees of wide distribution in India, except *T. travancorensis* which is confined to the evergreen forests of Travancore. *Combretum nanum* Buch.-Ham. ex D. Don, an undershrub of the outer Himalayas, W. Bengal, and the Central Provinces, bears terminal racemes of white, fragrant flowers which are  $\frac{1}{4}$  in. in diameter. The flowers of *Quisqualis indica* Linn., a large subscandent shrub which is commonly cultivated in gardens, are sweet-scented; they are  $2\frac{1}{2}$  in. long and  $1\frac{1}{2}$  in. across, white, soon turning red, and borne in axillary and terminal spikes.

Supplement

# AROMATIC PLANTS OF INDIA

## PART XII

BY

S. KRISHNA & R. L. BADHWAR

FOREST RESEARCH INSTITUTE, DEHRA DUN

FAMILY

46. Myrtaceae



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# AROMATIC PLANTS OF INDIA

## Family XLVI — MYRTACEAE\*

(Myrtle and Jamun Family)

THIS family comprises about 90 genera and 3,000 species of trees or shrubs which vary in size from small creepers to the giant eucalypts. It is found in warm regions, with Australia and tropical America as the chief centres. Its members are characterized by opposite and exstipulate leaves which, together with the young stems and parts of the flower and fruit, are usually copiously supplied with oil glands; for this reason the leaves are pellucid punctate and aromatic when crushed. The inflorescence is cymose and the flowers have numerous stamens and an inferior or half-inferior ovary.

This is an important family containing many useful trees and shrubs, which are either indigenous to India or have been introduced. The guava (*Psidium guajava* Linn.), rose apple [*Eugenia jambos* Linn., syn. *Syzygium jambos* (Linn.) Alst. and *Jambosa vulgaris* DC.], and the black plum or jamun [*Eugenia cumini* (Linn.) Druce, syn. *E. jambolana* Lam. and *Syzygium jambolanum* DC.] are some of the popular fruits. The fruit of *Rhodomyrtus tomentosa* (Ait.) Hassk. (hill guava, downy myrtle) of the Nilgiris is also edible. The dried unopened flower buds of *Eugenia caryophyllata* Thunb. [*E. aromatica* (Linn.) Baill., *Syzygium aromaticum* (Linn.) Merr. & Parry] constitute the well-known clove or *lavanga* which is one of the most widely used culinary spice; the annual demand for this spice is about 12,000 tons, most of which comes from Zanzibar and Pemba, and also to a large extent from Amboina, Madagascar, Penang, etc. The allspice or pimento (which is the rapidly dried unripe berry of *Pimenta officinalis* Lindl.) is another important culinary spice, which is in demand to the extent of 4,000 to 5,000 tons annually; it is supplied mostly by Jamaica.

A number of species of this family yield essential oils out of which the following are well known in commerce:—

Clove oil of commerce is obtained from the unopened flower buds of *Eugenia caryophyl-*

*lata* in a yield of 14 to 21 per cent. Its principal constituent is eugenol, which is usually present in the oil to the extent of 85 to 92 per cent. The bulk of the oil is employed for the extraction of pure eugenol which is required for the preparation of vanillin (artificial vanilla) — the famous flavouring agent. The flower stalks and leaves of cloves also yield about 5 per cent. of essential oils, which are known as clove-stem and clove-leaf oils in commerce. They are also rich in eugenol.

Allspice or pimento oil is obtained from the rapidly dried unripe berries (allspice, pimento) of *Pimenta officinalis* to the extent of 3 to 4.5 per cent. It contains 60 to 80 per cent. of eugenol. Like the oil from the berries, the leaf oil too is rich in eugenol.

The leaves of *Pimenta acris* Kostel. (Jamaica bayberry, wild clove) yield 1 to 1.5 per cent. of an essential oil known as bay oil or oil of myrcia, which is used in perfumery and in the preparation of bay rum. The principal constituents of this oil are eugenol and its methyl ether.

Cajuput oil of commerce is obtained from the leaves of *Melaleuca leucadendron* Linn. (most probably from its variety *minor* Duthie although it is stated to have been obtained from *M. leucadendron* type and other species of *Melaleuca*). It contains 45 to 55 per cent. or more of cineole, and is used in medicine and as an insect repellent. The Dutch East Indies, the principal producer of the oil, exported about 65 tons of the oil in 1937, whereas larger amounts were exported during previous years. The trade in this oil has declined to an extent, and this is due to the competition of Australian eucalyptus oils with a cineole content of over 70 per cent.

Niaouli oil or gomenol is obtained from *Melaleuca viridiflora* Soland. ex Gaertn. of New Caledonia, and in general resembles cajuput oil. A form of this species has recently been found to be a potential source of nerolidol and linalool. Nerolidol is in demand in high-class perfumery, but is

\* The Myrtaceae, as understood in this work, is exclusive of the family Lecythidaceae.

rather a rare ingredient in the vegetable kingdom. It can also be converted into farnesol, another rare ingredient.

Recently an essential oil from the leaves of the Australian *Melaleuca alternifolia* Cheel (narrow-leaved paperbark tea tree) has come into prominence for its germicidal value. It is obtained in a yield of 2 per cent., and contains cineole, terpinenes, and terpineol.

Myrtle oil of commerce, which finds use in soap industry, is obtained from the leaves of *Myrtus communis* Linn. (myrtle), a foreign plant. This species is cultivated in Indian gardens.

Essential oils from the Australian eucalypts are well known in commerce. They are generally grouped into (i) pharmaceutical or medicinal oils (sometimes called cineole oils from the name of the principal constituent), (ii) industrial oils (sometimes called phellandrene oils from the name of the principal constituent), and (iii) perfumery oils. Out of about 500 species and 138 varieties of *Eucalyptus*, less than 20 are being exploited at the present day for obtaining essential oils of commerce in Australia. These are: (a) *E. polybractea* R. T. Baker (blue mallee), *E. australiana* R. T. Baker & H. G. Smith (black peppermint), *E. dives* Schau., var. "C" (broad-leaved peppermint), *E. elaeophora* F. Muell. (applejack), *E. sideroxylon* A. Cunn. (red flowering ironbark), *E. leucoxylon* F. Muell. (white gum), *E. cneorifolia* DC. (narrow-leaved mallee), *E. viridis* R. T. Baker (green mallee), *E. dumosa* A. Cunn. ex Schau. (bull mallee), and *E. dives* Schau. (broad-leaved peppermint) — essential oils from all of these are used for medicinal purposes; (b) *E. phellandra* R. T. Baker & H. G. Smith\* (narrow-leaved peppermint), *E. dives* Schau., var. "A" (broad-leaved peppermint), *E. numerosa* Maiden (whitotop, river white gum), and *E. numerosa* Maiden, var. "A" (whitotop, river white gum) — essential oils from these are used for industrial purposes, such as in the "flotation" process for concentrating ore; and (c) *E. macarthuri* Deane & Maiden (Paddy's river box) and *E. citriodora* Hook. (lemon-scented gum). In addition, *E. odorata* Behr. (box tree or peppermint) and *E. smithii* R. T. Baker

(gully ash, white ironbark, whitetop) are also important species. During the year 1941-42, Australia exported 190,192 gallons of these oils. About 90 species of *Eucalyptus* have been introduced into India, but, of these, only *E. citriodora*, *E. dives*, *E. elaeophora*, *E. leucoxylon*, *E. macarthuri*, *E. sideroxylon*, and *E. smithii* are the important species. Even these do not appear to have attracted any attention in this country from point of view of the valuable essential oils, and only casual efforts have hitherto been made to cultivate them. If they have not been successfully cultivated at one place, no attempts have been made to try them at other places. On the other hand, attention has been mainly diverted to the cultivation of *E. globulus* Labill. (blue gum) in the Nilgiris, primarily for the supply of fuel and only incidentally for the distillation of essential oil from the leaves. The essential oil from this species, however, does not conform to the present British Pharmacopoeial standard. It is, therefore, suggested that proper species which yield valuable essential oils should be cultivated in India. In particular, special attention should be paid to the cultivation of shrubby species (the mallees), such as *E. polybractea*, *E. cneorifolia*, etc.

All the above-mentioned species yielding valuable essential oils are described in detail later. From among the other plants of the family yielding essential oils may be mentioned the following:—

Backhousia oil is obtained from the leaves of the Australian *Backhousia citriodora* F. Muell. in a yield of about 1 per cent. This essential oil is remarkable for the high proportion of citral (94 to 97 per cent.) which it contains. It is used in perfumery, to an extent, for its lemon-grass-like odour. It was also at one time a source of citral and a raw material for the manufacture of ionone, but is now only produced on a small scale since the oil of lemon grass is available at a cheaper rate. According to Penfold & Grant (1923. *J. roy. Soc. N.S.W.*, 57: 211) it has an extraordinarily high germicidal value, its R/W coefficient being 16. The leaves and branches of *B. myrtifolia* Hook. & Harv., another Australian plant, yield 0.22 to 0.76 per cent. of a pleasant-smelling oil which is entirely different from that of *B. citriodora*. This oil contains 75 to 80 per cent. of the phenol ether elemicin (Penfold, 1922. *Ibid.*, 56: 125). The dried leaves and terminal branches of *B. angustifolia* F. Muell. of

\* *Eucalyptus phellandra* was described by R. T. Baker & H. G. Smith in *Research on Eucalypts and their Essential Oils*, 1920: p. 280, but it has not been included in *Index Kewensis*. The Kew authorities, whose attention has been drawn to this omission, inform us that it will now be included in the eleventh Supplement.

Australia yield 1.05 per cent. of a pleasant oil containing as its principal constituent 75 per cent. of a substance which is probably a phenol allied to tasmanol and leptospermol; other constituents isolated from the oil are  $\alpha$ - and  $\beta$ -pinenes, cineole,  $\alpha$ -terpineol, a small quantity of a sesquiterpene and probably a solid lactone (Penfold, 1923. *Ibid.*, 57: 300). The leaves and terminal branchlets of another Australian species, *B. sciadophora* F. Muell., yield about 0.3 per cent. of an oil which consists in the main of pinene (80 to 85 per cent.), a sesquiterpene, a sesquiterpene alcohol, a phenol, and caprylic acid in the form of esters (Penfold, 1924. *Ibid.*, 58: 113).

The Australian species of the genus *Leptospermum* (tea trees) have been the subject of research in that country. They yield essential oils which differ considerably. Several species contain citral, while terpenes characterize some. The oils from different forms of *L. liversidgei* R. T. Baker & H. G. Smith are of interest. One form yields 0.25 to 3 per cent. of an oil containing 35 to 50 per cent. of citral and no citronellal, another gives about 0.55 per cent. of an oil containing 70 to 80 per cent. of citronellal and no citral, while the third form yields 0.6 to 0.8 per cent. of an oil containing 70 to 80 per cent. of citral — no citronellal? (Penfold, 1922. *J. roy. Soc. N.S.W.*, 56: 82). The second and third forms are rich sources of citronellal\* and citral, respectively. Further, the oil from the second form is identical in odour and cannot be distinguished except by chemical examination from the oil of *Eucalyptus citriodora* which is much richer in citronellal (containing 70 to 85 per cent.) than any other citronellal-containing oil. Another Australian species, *Leptospermum flavescens* Sm., yields from its leaves and branchlets about 0.8 per cent. of an essential oil containing dextrorotatory eudesmol, sesquiterpenes eudesmenene and aromadendrene,  $\alpha$ - and  $\beta$ -pinenes, a liquid phenol (leptospermol), and small amounts of citral and unidentified alcoholic bodies. According to Penfold (1921. *Ibid.*, 55: 170; vide also *Perfum. essent. Oil Rec.*, 1922, 13: 82), it has distinct commercial possibilities, especially in view of the large proportion of sesquiterpenes it contains. The sesquiterpenes have a fine cedarwood-like odour, and the oil could be

used for soap perfumery. The yellowish oil from the leaves of *L. odoratum* Cheel of Australia has a pleasant terpene and rose-like odour. According to Penfold (1920. *J. roy. Soc. N.S.W.*, 54: 197), it is obtained in a yield of 0.75 per cent. and consists essentially of the following terpenic compounds: dextrorotatory eudesmol, laevorotatory eudesmene, laevorotatory aromadendrene,  $\alpha$ -pinene,  $\beta$ -pinene, together with small amounts of a rose odour alcohol, butyric and acetic acid esters, and phenols, one of which is probably "tasmanol". Another Australian shrub *L. citratum* Challin., Cheel & Penf. (*L. flavescens* Sm., var. *citratum* Bailey & White) yields from its leaves and branchlets 1.73 to 1.85 per cent. of a pale-amber-coloured oil containing 90 per cent. of citral and citronellal in equal proportions with a small amount of an alcohol resembling geraniol or citronellol; a phenol and traces of aromadendrene are also present (Challinor, Cheel & Penfold, 1918. *Ibid.*, 52: 175). This oil occupies a position intermediate between those of *Eucalyptus citriodora* Hook. and *Backhousia citriodora* F. Muell., which contain the largest recorded quantities respectively of citronellal and citral. It possesses a strong, pleasant, modified lemon odour, suggestive of the principal constituents citronellal and citral. It may be useful as a soap perfume. In a recent publication, Penfold & Morrison (1946. *Aust. J. Pharm.*, 27: 723) state that this lemon-scented tea tree has three varieties; the type yields an oil containing 75 to 85 per cent. of a mixture of citral and citronellal, the variety "A" consists principally of  $\gamma$ -terpinene, while variety "B" contains geraniol and citronellal, together with a small percentage of citral and terpenes.

The essential oils of the several heath-like shrubby species of the Australian genus *Darwinia* are of interest in perfumery. The leaves of *D. fascicularis* Rudge yield 0.3 to 0.5 per cent. of an essential oil which contains 56 to 65 per cent. of geranyl acetate and 13 per cent. of geraniol (Baker & Smith, 1899. *J. roy. Soc. N.S.W.*, 33: 163). Those of *D. taxifolia* A. Cunn. yield 0.313 per cent. of an essential oil which contains *l*-pinene and 7.9 per cent. of an alcohol resembling linalool in odour (Baker & Smith, *loc. cit.*). The leaves and branchlets of *D. grandiflora* R. T. Baker & H. G. Smith (*D. taxifolia* A. Cunn., var. *grandiflora* Benth.) yield 0.35 per cent. of an essential oil containing *d*- $\alpha$ -pinene (as the principal constituent), a terpene, a sesquiterpene and its corresponding

\* St. Pfau (1925. *Perfum. essent. Oil Rec.*, 16: 183) has thrown a considerable doubt on the validity of the bisulphite method of estimating citronellal in the oil.

alcohol, and small quantities of isovaleric acid and amyl alcohol, along with the acetic ester of a new alcohol called darwinol  $C_{16}H_{34}O$  (Penfold, 1923. *Ibid.*, 57: 237; *Perfum. essent. Oil Rec.*, 1924, 15: 186). From another sample of this species Baker & Smith (1917. *J. roy. Soc. N.S.W.*, 50: 181) isolated 0.12 per cent. of an essential oil containing  $d$ - $\alpha$ -pinene (30 per cent.), geranyl acetate, and geranyl butyrate.

Homoranthus oil from the leaves of the Australian shrub *Homoranthus flavescens* A. Cunn. ex Schau. is the only Australian oil containing ocimene, which is present to the extent of about 80 per cent. It is obtained in a yield of 0.35 to 0.82 per cent., and the earlier fractions have a powerful odour of bananas due to traces of the amyl esters of acetic and butyric acids.  $d$ - $\alpha$ -Pinene and a sesquiterpene are also present in the oil (Penfold, 1923. *J. roy. Soc. N.S.W.*, 56: 197).

*Calyptanthus paniculata* Ruiz & Pav. of Peru yields an essential oil containing 62.5 per cent. of citral [Schimmel & Co., 1904 (April). *Rep.*: p. 95].

In addition to the above, a number of foreign plants belonging to the genera *Kuntzea*, *Angophora*, *Agonis*, etc., have also been investigated from point of view of their essential oils. Further information on these will be found in books on essential oils and other literature.

Little work has been done on the Indian myrtaceous plants, although they are known to contain essential oils. A systematic investigation of these is likely to yield useful results.

From among the constituents of essential oils reported from plants of the family may be mentioned the following:—

#### PARAFFINS

UNSATURATED HYDROCARBONS — Myrcene and ocimene.

AROMATIC HYDROCARBONS —  $p$ -Cymene and naphthalene.

MONOCYCLIC TERPENES —  $\gamma$ -Terpinene, phellandrene ( $\alpha$ - and  $\beta$ -),  $l$ -limonene, and dipentene ( $dl$ -limonene).

BICYCLIC TERPENES — Sabinene,  $\alpha$ -pinene ( $d$ -,  $l$ -, and  $i$ -),  $\beta$ -pinene, camphene, and fenchene.

BICYCLIC SESQUITERPENES — Cadinene,  $\alpha$ -caryophyllene,  $\beta$ -caryophyllene ( $d$ - and  $l$ -), and eudesmene.

UNSATURATED ALIPHATIC ALCOHOLS — Citronellol ( $d$ -,  $l$ -, and  $i$ -); geraniol, nerol, linalool, and nerolidol.

MONOCYCLIC ALCOHOLS — Menthol,  $\alpha$ -terpineol ( $l$ - and  $i$ -), piperitol, terpinenol-4, and isopulegol.

BICYCLIC ALCOHOLS — Myrtenol, pinocarveol fenchyl alcohol, and borneol.

BICYCLIC SESQUITERPENE ALCOHOL — Eudesmol.

SESQUITERPENE ALCOHOLS OF UNDETERMINED CONSTITUTION — Caryophyllol ( $C_{15}H_{26}O$ ) and globulol ( $C_{15}H_{26}O$ ).

UNNAMED SESQUITERPENE ALCOHOL ( $C_{15}H_{26}O$ ).

OTHER ALCOHOLS OF UNDETERMINED CONSTITUTION — Darwinol ( $C_{10}H_{18}O$ ).

PHENOLS AND PHENOLIC ETHERS — Eugenol, methyleugenol, chavicol,  $p$ -isopropylphenol (formerly australol), leptospermol, and other unnamed phenols and phenolic ethers.

ALIPHATIC ALDEHYDES — Citronellal ( $d$ - and  $l$ -) and citral.

ALICYCLIC ALDEHYDES — Phellandral ( $C_{10}H_{16}O$ ) and cryptal\* ( $C_{10}H_{16}O$ ).

HETEROCYCLIC ALDEHYDES

MONOCYCLIC KETONES — Menthone and piperitone.

UNIDENTIFIED KETONES

ACIDS AND ESTERS — Palmitic acid, etc.

UNIDENTIFIED LACTONE

OXIDE — Cineole (eucalyptol).

### 1. BAECKEA Linn.

(In honour of A. Baeck, a physician to the King of Sweden.)

This genus comprises 60 species of shrubs which are distributed from Australia to China. In India it is represented by one species, *B. virgata* Andr., which has been introduced into the Nilgiris from Australia. Its members have narrow, pointed leaves with many pellucid glands, 5-merous axillary flowers, and a capsular fruit. Essential oils have been reported from the leaves of many species.

The leaves of *B. frutescens* Linn., a foreign shrub or a small tree, are used in some Pacific islands for the preparation of a refreshing herbal tea, which is given in fevers and in cases of lassitude. In Annam, the flowers are said to be used for making the herbal tea. In Indo-China, the leaves are kept among clothes as an insect repellent. They are reported to yield a pale-yellow essential oil with the smell of lavender (Burkill, 1935. *Dictionary of Economic Products of Malay Peninsula*, 1: 282). Its sp. gr.<sup>20</sup> is 0.883,

\* This is now known as  $l$ -cryptone (Penfold, 1949. *Private communication*).

and it contains a stearoptene (Gildemeister & Hoffmann).

From among the constituents of essential oils isolated from the leaves or leaves and tender branches of other species of *Baeckea* may be mentioned:  $\alpha$ - and  $\beta$ -pinenes, cineole, sesquiterpenes and their alcohols, such as eudesmol, a crystalline stearoptene, cymene, phenols and their ethers, aldehydes, alcohols, and acids, such as isobutyric, valeric and isovaleric, and their esters.

### ***Baeckea virgata* Andr.**

(Australian May, Tea Tree of Australia)

This is usually a tall, erect, and loosely branched shrub with slender twigs. It attains a height of 10 to 12 ft., but is rarely low and diffuse. The leaves have numerous pellucid glands, which are raised on the lower side when dry. The flowers are small and arranged in umbels in the axils of upper leaves. This shrub is a native of Australia from Victoria to Queensland, and is introduced into the Nilgiris.

The leaves and twigs of this plant, from Queensland, have been reported to yield on steam distillation about 1 per cent. of an essential oil having the following characteristics: sp. gr.  $^{15.50}$  0.9021,  $[\alpha] +18^\circ$ ,  $n^{20}$  1.4742, acid val. 1, and ester val. after acetylation 17. The oil contains about 50 to 60 per cent. *d*- $\alpha$ -pinene, about 30 per cent. cineole and aromadendrene (?), a sesquiterpene alcohol  $C_{15}H_{24}O$  (b.p.  $105^\circ$  to  $107^\circ$  at 4 to 5 mm.), and traces of an alcohol  $C_{10}H_{18}O$  (b.p.  $220^\circ$ ), presumably pinocarveol (Jones & White, 1935. *Proc. roy. Soc. Qd.*, 42: 49).

### **2. CALLISTEMON R. Br.**

(From the Greek *kallos* — beauty, and *stemon* — a thread; referring to the beautiful bottle-brush-like appearance of flower spike.)

This genus comprises 12 Australian species (bottle brushes) of trees and shrubs, often with willowy foliage and small bright-red or crimson flowers which are grouped together in pendulous spikes; the spikes resemble somewhat a bottle brush — hence the popular name. The axis of the inflorescence grows on beyond the flowers and continues to produce leaves. The small dry fruits remain for a long time clustered round the branches far below the terminal leaves. In India it is represented by 8 species, which are cultivated in gardens as ornamental flowering plants.

The leaves of *C. salignus* Sweet, which is cultivated in Dehra Dun, Saharanpur, Calcutta, Bihar, etc., emit a myrtle-like odour, when bruised. The leaves of another introduced species, *C. linearis* DC., has also aromatic leaves. No essential oil appears to have been distilled from either of these plants. Of all the introduced species, only *C. lanceolatus* Sweet and *C. viminalis* (Soland.) Cheel have been investigated from the point of view of essential oils, and these are described below in detail.

#### **1. *Callistemon lanceolatus* Sweet**

(Bottle Brush)

This is a small tree with rough greyish-brown bark, which is deeply cleft vertically into narrow ridges. The filaments of the stamens are about  $\frac{3}{4}$  in. long, deep crimson, and the anthers dark. It is a native of Australia, and is frequently cultivated in Indian gardens, as at Dehra Dun.

The leaves and branches of Australian specimens are reported to yield, on steam distillation, 0.06 to 0.22 per cent. of an essential oil having the following characteristics: sp. gr. 0.889 and 0.907,  $[\alpha] -0.52^\circ$  and  $-0.5^\circ$ ,  $n$  1.4716 and 1.4668, ester val. (hot  $1\frac{1}{2}$  hours) 9.1 and 5.5, and the same after acetylation 30.3. The oil so obtained contains cineole (up to 53 per cent.), dipentene, limonene,  $\alpha$ -terpineol, sesquiterpene, and phenol (0.5 per cent.). This oil, which is of pale lemon-yellow colour, has a pronounced odour of cineole, and is very similar to those obtained from the cineole group of the eucalypts. It is free from the objectionable volatile aldehydes and phellandrene, and would have been of considerable value as a medicinal oil, only if the yield were not so low (0.2 per cent.) in comparison with 2 to 3 per cent. obtained from some of the species of *Eucalyptus* (Penfold, 1923. *J. roy. Soc. N.S.W.*, 57: 131).

#### **2. *Callistemon viminalis* (Soland.) Cheel**

(Bottle Brush)

This is a small evergreen tree with linear-lanceolate leaves and crimson flowers in spikes. It is a native of Queensland and New South Wales, and is cultivated in Indian gardens, as at Dehra Dun.

Penfold (1923. *J. roy. Soc. N.S.W.*, 57: 131) obtained by steam distillation 0.2 per cent. of an essential oil from the leaves

FIG. 45 — *Callistemon lanceolatus* Sweet

and branches. This oil had the following characteristics :

	NEW SOUTH WALES OILS		QUEENSLAND OILS	
	1	2	1	2
Specific gravity	0.889	0.898	0.920	0.913
Optical rotation	+14.1°	+12.8°	+0°	-1°
Refractive index	1.4677	1.4678	1.4609	1.4634
Ester val. (hot 1½ hr.)	17.6	18.9	5.5	7.3
Ester val. after acetylation	38.8	34.1	21.5	20.6
			70 per cent.	
Solubility in 80 per cent. alcohol	1 in 3.8	1 in 5	1 in 1	1 in 1.4

The main constituents of the oil from New South Wales were cineole (about 30 per cent.), *d*- $\alpha$ -pinene, amyl alcohol, a sesquiterpene, and a phenol (2 per cent.). The Queensland oil contained cineole (60 to 80 per cent.), dipentene, limonene, terpineol, a sesquiterpene, and a phenol (0.5 per cent.).

This oil, like that from *C. lanceolatus* Sweet, closely resembles those obtained from the cineole group of the eucalypts. It is free from the objectionable volatile aldehydes and phellandrene and, according to Penfold (*loc. cit.*), would have been of considerable value as a medicinal oil, were it not for the poor yield (0.2 per cent.).

### 3. EUCALYPTUS L'Hérit.\*

(From the Greek *eu* — well, and *kalypto* — I cover; in allusion to the operculum or lid which covers the calyx until the stamens are fully developed.)

This is a large genus of over 500 species and 138 varieties of evergreen tall trees or dwarfed shrubs. Most of the trees are popularly called gum trees in Australia, although the gummy substance that exudes from them is not a gum but an astringent, tanniferous substance called kino which is used in the tanning of hides. This "gum" sometimes runs down the trunk in such quantities that the species are called bloodwood in consequence. The shrubs are known as mallees, and these have a bunchy growth of small stems springing from a root crown. Eucalypts are confined to Australia, Tasmania, New Guinea and other neighbouring islands, where they provide characteristic features to the scenery. In their natural habitat they are distributed widely over the continent of Australia from the sandy regions of low rainfall where mallees abound to the coast and mountain regions where the tall, luxuriant trees attain gigantic dimensions. In fact, they probably constitute three fourths of the whole vegetation of that continent.

Eucalypts are of great economic importance from the point of view of timber, essential oils and tannins, and various

\* We are grateful to Mr. A. R. Penfold, Director, and his colleagues of the Museum of Technology and Applied Science, Sydney, for reading the manuscript on eucalypts and making useful suggestions.



species have been brought into cultivation all the world over, particularly on account of their beauty. All the species introduced into India have the tree habit, and many of them grow quite tall.

The species of *Eucalyptus* are difficult of identification, and consequently there is a difference of opinion with regard to their number. All the species are more or less aromatic and contain oil glands in the rigid leaves. The leaves on young plants and young coppice shoots are generally larger, opposite, sessile and horizontal, and are often of a different shape from the normal leaves of the adult trees; the latter are usually alternate, petiolate, lance-shaped, curved like a sickle, and hang vertically. The veins of leaves are characteristic, two of them called intramarginal veins being present in most of the species running along the margin, slightly removed from it. The nature of the lateral veins springing from the midrib is characteristic, and it has been found to be an indication of the constituents of the oil in a general way. For example, an obtuse feather-like venation is indicative of pinene as the principal constituent and of low yield, as in *E. corymbosa* Sm.; a lateral venation with a marginal vein represents a slightly higher yield with cineole and pinene as constituents, as in *E. globulus* Labill., etc. Such a generalization has been of some practical utility. The bark is variable in its nature and texture, being either rough, stringy, furrowed or smooth; it often peels off in large flakes. The character of the bark is often helpful in distinguishing the species.

Eucalypts are remarkable for their rapid growth. Some of the species reach a gigantic size, and are among the tallest trees in the world, specimens of the blue gum (*E. globulus* Labill.), karri (*E. diversicolor* F. Muell.), woolly butt (*E. marginata* Sm.), stringybark (*E. obliqua* L'Hérit.), and peppermint gum (*E. regnans* F. Muell., formerly included under *E. amygdalina* Labill.) having been stated to reach a height of over 400 ft. They have been supposed to be useful if planted in malarial regions, and this is one of the reasons for their introduction into many countries. Investigations in this respect, however, "appear to show that the only beneficial influence is the removal of some excess water by transpiration of moisture from the leaf surface and a limited antasthmatic action due to escape of cineol[e] into the air and its conversion into ozone"

(*Webster's New International Dictionary*, 1927, p. 755).

This Australian genus presents a series of woods whose variety in terms of physical properties and qualities is probably unequalled in a single genus elsewhere in the world. Among the species which yield commercial timber may be mentioned: *E. longifolia* Link & Otto (woolly butt), *E. maculata* Hook. (spotted gum), *E. marginata* Sm. (jarrah), *E. microcorys* F. Muell. (tallow-wood), *E. paniculata* Sm. (white ironbark), *E. patens* Benth. (blackbutt), *E. pilularis* Sm. (blackbutt), *E. resinifera* Sm. (Australian red mahogany) and *E. salubris* F. Muell. (gimlet gum).

*E. gunnii* Hook. f., called cider gum in Tasmania, yields a refreshing drink from wounds made in the bark in the spring.

Another important product is the eucalyptus oil.\* The oils obtained from different species are generally grouped into (i) pharmaceutical or medicinal oils (sometimes called cineole oils from the name of the principal constituent), (ii) industrial oils (sometimes called phellandrene oils from the name of the principal constituent), and (iii) perfumery oils. These oils and their ingredients have been applied in many ways.

The original uses of eucalyptus oils were medicinal, and the first to be used for this purpose was *E. piperita* Sm. (Sydney peppermint). The oil from this species was found to be efficacious against colicky pains (White; *vide* Penfold, 1935. *Aust. J. Pharm.*: p. 29). Later on several other species, such as *E. globulus* Labill. (blue gum), *E. oleosa* F. Muell. (red mallee, water mallee), and *E. cneorifolia* DC. (narrow-leaved mallee), appear to have been distilled for commercial purposes in the early eighties of the last century. At one time *E. globulus* was the favourite with *E. amygdalina* Labill. (black peppermint) a good second. According to Penfold (*loc. cit.*), the former has since received much greater attention than any other species of *Eucalyptus*. In India, too, although quite a large number of eucalypts

\* The term eucalyptus oil is rather vague, and has no precise meaning. Although it is often supposed to be a standard material consisting mostly or wholly of cineole (eucalyptol), the odour of which is familiar to most people who have used it as a cure for cold or influenza, various species have their own distinctive oils. Each of these oils is a complex mixture of various constituents, and their odours cover a wide range, including turpentine, cineole, peppermint, lemon, and geranium as dominant notes.



have been introduced from as early as 1843, chiefly on the Nilgiris, it is only from *E. globulus* that the oil is distilled for commercial purposes. Indeed, this is the only species which has been planted largely. This industry is, however, confined to the Nilgiris where the eucalypts, chiefly *E. globulus*, cover an area of about 2,550 acres and about 22,000 gallons of the oil are produced annually (Anon., 1940. *Indian J. Pharm.*: p. 183).

So far as the eucalyptus oil industry of Australia is concerned, the position has completely changed. Practically all the above-mentioned species, except *E. cneorifolia*, have been superseded by those giving larger yields of oil. The investigations of Baker & Smith (1920. *A Research on the Eucalypts and their Essential Oils*) have shown that other species, such as *E. polybractea* R. T. Baker (blue mallee), *E. australiana* R. T. Baker & H. G. Smith (black peppermint), *E. phellandra* R. T. Baker & H. G. Smith (narrow-leaved peppermint), etc., give higher yields of oil of equal or better quality. It is from these species, therefore, that the present-day eucalyptus oils of commerce are won in Australia, and not from species like *E. globulus*.

Out of over 500 species and 138 varieties of *Eucalyptus*, about 180 types, including varieties, have so far been investigated for essential oils. Among these, less than 20 are being exploited at the present day for obtaining eucalyptus oils of commerce in Australia. The following is a list of these (Penfold, 1935. *Aust. J. Pharm.*: p. 29; Penfold & Morrison, 1944. *Commercial Eucalyptus Oils. Bull. tech. Mus., Sydney*, No. 2: p. 1-36).

The average yield of oil furnished by the mature leaves and terminal branchlets ranges from about 3.5 per cent. in *E. australiana* and *E. phellandra* to 0.6 per cent. in *E. corymbosa*, and dwindles to mere traces in still poorer species. The occurrence of physiological forms in many species is, however, of considerable economic importance. This matter has been dealt with by Penfold (1935. *Aust. J. Pharm.*: February, 28). Writing about *E. dives* he states, "The observation was made of two trees growing together, indistinguishable from one another by both botanist and bushmen, but each containing a different essential oil.... It was therefore apparent that several varieties or forms of *Eucalyptus dives* existed distinguishable only by chemical means." Chemically speaking,

NAME	PRINCIPAL CONSTITUENTS %
<b>Medicinal Oils</b>	
<i>E. polybractea</i> R. T. Baker (blue mallee)	Cineole 80 to 85
<i>E. australiana</i> R. T. Baker & H. G. Smith (black peppermint)	Cineole 70 to 75
<i>E. dives</i> Schau., var. "C" (broad-leaved peppermint)	Cineole 70 to 75
<i>E. elaeophora</i> F. Muell. (applejack)	in admixture known as applejack and ironbark
<i>E. sideroxylon</i> A. Cunn. (red flowering ironbark)	
<i>E. leucoxylon</i> F. Muell. (white gum)	
<i>E. cneorifolia</i> DC. (narrow-leaved mallee)	Cineole 70
<i>E. viridis</i> R. T. Baker (green mallee)	Cineole 70 to 80
<i>E. dumosa</i> A. Cunn. ex Schau. (bull mallee)	Cineole 70
<i>E. dives</i> Schau., type (broad-leaved peppermint)	Piperitone 40 to 50 and phellandrene 40
<b>Industrial Oils</b>	
<i>E. phellandra</i> R. T. Baker & H. G. Smith (narrow-leaved peppermint)	Phellandrene and cineole
<i>E. dives</i> Schau., var. "A" (broad-leaved peppermint)	Phellandrene
<i>E. numerosa</i> Maiden (whitetop, river white gum)	Phellandrene and piperitol
<i>E. numerosa</i> Maiden, var. "A" (whitetop, river white gum)	Piperitone and phellandrene
<b>Perfumery Oils</b>	
<i>E. macarthurii</i> Deane & Maiden (Paddy's river box)	Geranyl acetate, geraniol, and eudesmol
<i>E. citriodora</i> Hook. (lemon-scented gum)	Citronellal

the difference lay in the proportion of piperitone, phellandrene, and cineole content. The type species contained 46 to 53 per cent. piperitone and about 40 per cent. phellandrene, whereas "form A" had 5 per cent. piperitone and 75 to 80 per cent. phellandrene, "form B" 10 to 20 per cent. piperitone, 25 to 45 per cent. cineole, and a fair amount of phellandrene, and "form C" less than 5 per cent. piperitone, 60 to 70 per cent. cineole, and no phellandrene. Similar physiological forms have been described in many other species. Recently, the discovery of a form of *E. macarthurii*, which yields a high percentage of essential oil, has been the object of much comment in the press. The reference in the lay press that this strain yields ten times more oil than the type is incorrect (Penfold, 1949. *Private communication*), but the seedlings have been raised from this high-yielding strain for further study [Division of Wood Technology, Forestry Commission of N.S.W., January, February, March, 1948, 3 (1, 2, 3): 10].

The work of Carter & Read (1925. *J. Soc. chem. Ind., Lond.*, 44: 525T), however, indicates that the oil from different species contains a certain selection of the chemical

constituents in fairly definite proportions and is thus distinctive for the species. Their results also indicate that, although eucalyptus oil derived from the same species cultivated outside its natural Australian habitat may show appreciable difference in physical constants, it nevertheless contains sensibly identical amounts of the main components of commercial value. In view of the findings of Penfold (*loc. cit.*) about the existence of physiological forms, the conclusion of Carter & Read must be understood to mean that the composition of the leaf oils appears to remain unchanged, not within the range of a species, but of the same physiological forms, outside their natural habitat. This is important for those interested in the introduction of commercial species in various countries. The examples of oils from *E. globulus* and *E. citriodora* cultivated in different parts of the world confirm this view.

*Introduction into and cultivation in India* — It is probable that the earliest attempts to grow eucalypts in India were those made in the Nilgiris in 1843, when *E. globulus* was planted at Ootacamund. This was mainly with the object of finding some species which could be cultivated there for the supply of fuel. A large number of other species have since been tried all over India, both on the hills and in the plains. According to Troup (1921. *Silviculture of Indian Trees*), some species or other has been found which is suitable for almost any climate in India with the possible exception of (a) very moist tropical regions, where the seedlings damp off in spite of all precautions, and (b) elevations above the winter snowline, where snowbreak is to be feared. The experimental data so far collected indicate that, for low elevations in India, species from the tropical and warm parts of Queensland and adjacent northern regions are suitable, whereas those from Tasmania and the southern parts of Australia are likely to be successful at higher elevations.

A good deal of attention has hitherto been paid to the cultivation of eucalypts in India, but in spite of this there has been much confusion. In this connection it must be emphasized that, for the correct identification of eucalypts, complete botanical specimens should ordinarily include primordial as well as adult leaves, flowers, fruits, and bark. Since these are not available at one time, sufficient care should be taken to ensure that the specimens are collected from the same tree.

As elsewhere in the world, the close resemblance of a number of eucalypts has been the cause of much error in the identification of various species introduced into India. It is, therefore, feared that information on their cultivation might not be as accurate as desirable. Subject to this doubt, the following is the list of the species so far cultivated in India. The places where they have been cultivated and the amount of success achieved are also briefly given:—

1. *E. acmenioides* Schau. (white mahogany). A fairly tall, straight-growing tree. A few specimens in the Nilgiris; being tried in the plains of N. India, but appears not likely to succeed.
2. *E. alpina* Lindl. A stunted tree or shrub.
3. *E. amygdalina* Labill. (black peppermint). A small to large tree. Not a success in the plains of N. India; does well at 5,000 ft. in Simla Hills.
4. *E. andrewsi* Maiden (New England peppermint). A tall tree.
5. *E. baileyana* F. Muell. (rough stringybark). A large tree.
6. *E. bicolor* A. Cunn. ex Hook. (red box). A small to moderately tall tree. Doing well in Simla Hills at 4,000 ft. Does not grow well in plains with heavy rainfall; doing well in Delhi on well-drained sandy soil.
7. *E. bosistoana* F. Muell. (ribbon box). A tall tree. Has grown slowly during early years in plains of N. India.
8. *E. botryoides* Sm. (bastard mahogany, bangalay). A medium-sized to large tree. Grows fairly well in the Nilgiris; not succeeded in the Andaman mangroves; establishes well in Dehra Dun.
9. *E. calophylla* R. Br. (red gum). A moderate-sized to large tree. Grows in the Nilgiris; not promising below Simla Hills; failed in plains of N. India.
10. *E. capitellata* Sm. (brown stringybark). A moderate-sized to tall tree. Grown to a limited extent in the Nilgiris; successful in plains of N. India.
11. *E. cinerea* F. Muell. ex Benth. (Argyle apple). A moderate-sized tree, not promising in plains of N. India; some trees are growing in the Nilgiris.
12. *E. citriodora* Hook. (citron-scented gum, lemon-scented gum). A tall tree. Much planted in the plains of N. India and in the submontane tract; not capable of resisting severe frost or excessive drought. Grown to some extent in the Nilgiris and

FIG. 46 — *Eucalyptus citriodora* Hook.

- in Mercara in Coorg, where it is one of the most promising species so far tried.
13. *E. cladocalyx* F. Muell. ; syn. *E. corynocalyx* F. Muell. (sugar gum). A shrub or a tree, not successful in Simla Hills and plains of N. India.
  14. *E. coccifera* Hook. f. A small tree, unsuccessful in plains of N. India.
  15. *E. coriacea* A. Cunn. ex Schau. ; syn. *E. pauciflora* Sieb. ex Spreng. (white gum, scribbly gum). A moderate-sized tree. Grows moderately well in Simla Hills at 4,000 to 6,000 ft. ; failed in plains of N. India.
  16. *E. cornuta* Labill. (yate gum). A moderate-sized to large tree. Failed in plains of N. India ; likely to do well at 4,000 ft.
  17. *E. corymbosa* Sm. (bloodwood). A fairly tall tree. Grows moderately well in Simla Hills above 6,000 ft. ; failed in plains of N. India.  
*E. corynocalyx* F. Muell. ; see *E. cladocalyx* F. Muell.
  18. *E. crebra* F. Muell. (narrow-leaved ironbark). A moderate-sized to large tree. Grown to a limited extent in the Nilgiris ; done moderately well in Simla Hills from 4,000 ft. upwards ; occasionally grown in plains of N. India with variable success ; proved successful in Dehra-Dun.
  19. *E. dealbata* A. Cunn. ex Schau. (white gum, mountain gum). A fair-sized tree often seen in Kumaon at 5,000 to 6,000 ft. ; does not thrive in the plains.

20. *E. deanei* Maiden. A large tree, not successful in plains of N. India; worth trying again.
21. *E. deglupta* Blume. Grows fairly well at Dehra Dun, but is slightly affected by severe frost.
22. *E. delegatensis* R. T. Baker (silver-topped mountain ash, white ash). A tall tree.
23. *E. diversicolor* F. Muell. (karri). A very large tree, failed in plains of N. India.
24. *E. dives* Schau. (peppermint, broad-leaved peppermint). A moderate-sized tree. Shown little promise in plains of N. India.
25. *E. drepanophylla* F. Muell. ex Benth. Has done well in Kangra district, Lachiwala, and Dehra Dun; unaffected by monsoon.
26. *E. elaeophora* F. Muell. (bundy, half box). A small to moderate-sized tree. Failed in plains of N. India; does well in Simla Hills at about 5,000 ft., but becomes crooked and is easily damaged by snow.
27. *E. eugenioides* Sieb. ex Spreng. (white stringybark). A moderate-sized to fairly tall tree. Failed in plains of N. India. In Simla Hills sensitive to frost, but likely to do well between 4,000 and 5,000 ft.
28. *E. eximia* Schau. (white or yellow bloodwood). A small to moderate-sized tree. Done well in Simla Hills below 6,000 ft.; failed in plains of N. India.
29. *E. ficifolia* F. Muell. (red flowering gum). A small bushy tree, extensively grown in the Nilgiris; failed in plains of N. India.
30. *E. foecunda* Schau. A shrub or small tree, occasionally grown in the Nilgiris; does not thrive in plains of N. India.
31. *E. globulus* Labill. (blue gum). A very tall tree. Succeeded at or above 4,000 ft. in Simla Hills, Abbottabad, Kumaon, Darjeeling, and Shillong. Best results obtained in the Nilgiris where it is planted extensively. Liable to breakage where snowfall is heavy.
32. *E. gomphocephala* DC. (tuar). A moderate-sized to fairly tall tree. Does not thrive in plains of N. India and in Simla Hills.
33. *E. goniocalyx* F. Muell. (mountain gum). A large fast-growing tree. Succeeded moderately well in Simla Hills between 4,000 to 5,000 ft.; failed in plains of N. India.
34. *E. gunnii* Hook. f. (swamp gum, cider gum). A small or moderate-sized tree cultivated in the Nilgiris; failed in plains of N. India; does well at 4,000 to 6,000 ft. in Simla Hills.
35. *E. haemastoma* Sm. (scribbly gum, white gum). A small or moderate-sized tree, tried without success in the plains of N. India.
36. *E. hemiphloia* F. Muell. (grey box, white box). A fairly large tree. Grows fairly well in plains of N. India; may succeed up to 4,000 ft. in hills.
37. *E. leucoxydon* F. Muell. (Victorian ironbark, white gum). A moderate-sized tree grown in the Nilgiris where it attains only a small size; grows moderately well in Simla Hills at 5,000 ft., but probably will fare better at 2,000 to 4,000 ft.
38. *E. linearis* Dehnh. (white peppermint). A small tree growing fairly well at about 5,000 ft. in the Himalayas; suffers from frost and snow at higher elevations.
39. *E. longifolia* Link & Otto (woolly butt). A large tree grown in the Nilgiris; done well in Simla Hills at 4,000 to 5,000 ft., but not suitable for plains.
40. *E. macarthurii* Deane & Maiden (Paddy's river box, Camden woolly butt). A moderate-sized tree tried without success in plains of N. India.
41. *E. macrandra* F. Muell. ex Benth. A shrub or small tree, tried in plains of N. India but has not shown much promise.
42. *E. macrocarpa* Hook. A large ornamental shrub which has not shown much promise in plains of N. India.
43. *E. macrorhyncha* F. Muell. (red stringybark, Victorian stringybark). A moderate-sized tree grown in the Nilgiris; unsuccessful in Simla Hills and in plains of N. India, but further trials at about 5,000 ft. may be successful.
44. *E. maidenii* F. Muell. (blue gum). A tall, straight tree which has done well in Simla Hills between 4,000 to 7,000 ft., but failed at lower elevations. It is frost-hardy.
45. *E. marginata* Sm. (jarrah). A large tree tried in several places, but has not so far established anywhere.
46. *E. melanophloia* F. Muell. (silver-leaved ironbark). A small to moderate-sized tree which has done well in the plains of N. India and appears to be very drought-resistant.

FIG. 47 — *Eucalyptus globulus* Labill.

47. *E. melliodora* A. Cunn. ex R. T. Baker & H. G. Smith (yellow box). A moderate-sized tree; done fairly well below 6,000 ft. in Simla Hills and in plains of N. India.
48. *E. microcorys* F. Muell. (tallow-wood). A large tree grown to a small extent in the Nilgiris; done moderately well in Simla Hills below 4,000 ft., and proved successful in early stages at Saharanpur in the plains.
49. *E. microtheca* F. Muell. (coolabah, tangoon). A small to moderate-sized tree tried with success in plains of N. India; appears drought-resistant, but does best with subsoil moisture.
50. *E. miniata* A. Cunn. ex Schau. A very ornamental tree with red flowers. Said to be growing in the Nilgiris.
51. *E. muelleriana* Howitt (yellow stringy-bark). A moderate-sized to large tree; not shown any promise in Simla Hills and in plains of N. India.
52. *E. obliqua* L' Hér. (stringybark, messmate). A very tall straight tree which, after *E. globulus*, is one of the commonest species in the Nilgiris; failed at Saugor in the Central Provinces, in Simla Hills and in plains of N. India.
53. *E. occidentalis* Endl. A moderate-sized tree, attaining fairly large dimensions in favourable localities, but often little more than a shrub. Tried in plains of N. India, but not promising.
54. *E. ovata* Labill. A small to moderate-sized tree; done moderately well in Simla Hills from 4,000 to 6,000 ft., but failed in plains.

55. *E. paniculata* Sm. (white ironbark). A moderate-sized to large tree grown successfully to a limited extent in the Nilgiris, Simla Hills below 4,000 ft., at Saharanpur, and in Changa Manga irrigated plantation in the Punjab.
56. *E. patentinervis* R. T. Baker (half mahogany). A good sized tree growing fairly well in the Punjab plains. *E. pauciflora* Sieb. ex Spreng.; see *E. coriacea* A. Cunn. ex Schau.
57. *E. pilularis* Sm. (blackbutt). A large tree. Grown sparingly in the Nilgiris; has done well in earlier stages at Mercara in Coorg. Grows well in Simla Hills at about 5,000 ft., but not a success in plains.
58. *E. piperita* Sm. (Sydney peppermint). A small or moderate-sized tree not successful in Simla Hills and in the plains of N. India.
59. *E. planchoniana* F. Muell. (stringybark). A small to moderate-sized tree not successful in Simla Hills above 4,000 ft.
60. *E. platypus* Hook. A small tree unsuccessful in plains.
61. *E. polyanthemus* Schau. (red box). A small to moderate-sized tree. Successfully grown at 4,000 ft. in Simla Hills; moderately successful in plains.
62. *E. populifolia* Hook. (bimil box, poplar-leaved box). A small tree which grows slowly in plains of N. India.
63. *E. propinqua* Deane & Maiden\* (grey gum of the north coast, New South Wales). A large tree showing promise in plains of India.
64. *E. ptychocarpa* F. Muell. A moderate-sized ornamental tree with red flowers. Grown to a small extent in the Nilgiris.
65. *E. pulverulenta* Sims. A tall shrub or a small scraggy tree. Grown to a small extent in the Nilgiris.
66. *E. punctata* DC. (grey gum). A small to moderate-sized tree doing well in Simla Hills at lower elevations. Grows well at Dehra Dun. Successful in early stages at Mercara, Coorg. Fairly successful in plains, but does not appear suitable for very hot localities.
67. *E. redunca* Schau. (white gum). Shrub or a small tree grown on a small scale in the Nilgiris. Not successful at lower elevations in hills of N. India.
68. *E. regnans* F. Muell. (mountain ash, giant gum). A very large tree. Succeeded well in Simla Hills at 4,000 ft. on good soil, being the only species other than *E. coriacea* that withstands snow; failed in plains.
69. *E. resinifera* Sm. (Australian red mahogany). A large tree. A few specimens exist in the Nilgiris. Moderately successful below 4,000 ft. in Simla Hills, but failed in plains.
70. *E. robusta* Sm. (swamp mahogany). A moderate-sized to large tree of which there are a few specimens in the Nilgiris. Grown in various parts of the plains of N. India and in the Sub-Himalayan tract. Succeeds only in moist situations. Failed in mangrove swamps in the Andamans.
71. *E. rostrata* Schlecht. (Murray red gum). A tall tree successfully tried in Simla Hills up to 5,000 ft.; grows well in the plains, particularly where subsoil water is not too deep.
72. *E. rubida* Deane & Maiden (candlebark). A moderate-sized tree. Not shown any promise in plains of N. India.
73. *E. rudis* Endl. (flooded gum, swamp gum). A small to moderate-sized tree proved fairly successful in Simla Hills below 6,000 ft. Grows rapidly in early years in plains of N. India and in the Sub-Himalayan tract, even on saline soil. Thrives on swampy ground, also in comparatively dry land; best results under irrigation or where subsoil water level is near the surface.
74. *E. saligna* Sm. (Sydney blue gum). A large tree grown in the Nilgiris. Done moderately well in Simla Hills well above 4,000 ft. Grown successfully to small extent in plains of N. India.
75. *E. salmonophloia* F. Muell. (salmon-coloured gum). A moderate-sized to fairly large tree. Tried in Simla Hills without definite results. Not promising in plains.
76. *E. salubris* F. Muell. (gimlet gum). A rather small tree, not successful in plains.
77. *E. siderophloia* Benth. (Sydney ironbark). A moderate-sized tree growing fairly well in the Nilgiris. Occasional trees in plains of N. India, but not succeeded everywhere.

\* *Eucalyptus propinqua* was described by Deane & Maiden in *Proc. Linn. Soc. N.S.W.* ser. 2, 10; 1895: p. 541, tab. 43, but it has not so far been included in *Index Kewensis*. The Kew authorities, whose attention has been drawn to this omission, inform us that it will now be included in the eleventh Supplement.

78. *E. sideroxylon* A. Cunn. (red flowering ironbark). A moderate-sized to large tree. Done well in Kumaon Hills. Grown at Abbottabad where it survives frost. Done moderately well in Simla Hills between 4,000 and 6,000 ft. Not suitable for plains.
79. *E. sieberiana* F. Muell. (mountain ash). A large tree grown to some extent in the Nilgiris. Appears promising in Simla Hills between 5,000 and 6,000 ft.
80. *E. smithii* R. T. Baker (gully ash, white ironbark, whitetop). A moderate-sized to large tree tried in plains of N. India but not shown promise.
81. *E. stellulata* Sieb. ex DC. (lead gum, black sally). A moderate-sized tree.
82. *E. stricta* Sieb. ex Spreng. A shrubby species which has not shown promise in plains of N. India.
83. *E. stuartiana* F. Muell. ex Miq. (apple box). A moderate-sized tree grown in the Nilgiris. Done moderately well in Simla Hills, particularly between 6,000 to 8,000 ft., but has failed in the plains.
84. *E. tereticornis* Sm. (forest red gum). A tall tree grown in many parts of India. In Simla Hills done well below 4,000 ft. and moderately well from 4,000 to 6,000 ft. Successful in the Sub-Himalayan tract. One of the best species in the plains of N. India. Fairly successful in the Nilgiris.
85. *E. torrelliana* F. Muell. An ornamental tree. Has shown good growth in Dehra Dun.
86. *E. trachyphloia* F. Muell. (bloodwood). A tall tree. Tried in plains of N. India, but has not shown much promise.
87. *E. umbra* R. T. Baker (stringybark, white mahogany). A moderate-sized to fairly tall tree tried without success in N. India.
88. *E. urnigera* Hook. f. (urn gum). A small to moderate-sized tree quite successful in plains of N. India.
89. *E. viminalis* Labill. (manna gum). A large tree. Likely to be one of the best species for Simla Hills on good soils between 5,000 to 6,000 ft.; unsuccessful in plains.
90. *E. virgata* Sieb. ex DC. A moderate-sized tree which has been tried without success in plains of N. India.

It will thus be seen that of the thirteen species from which the present-day commercial oils of eucalypts are won in Australia,

only *E. citriodora*, *E. dives*, *E. elaeophora*, *E. leucoxylon*, *E. macarthurii*, and *E. sideroxylon* have been tried in India. Of these, *E. citriodora* grows well in the plains of N. India and submontane tract and in Coorg; *E. dives* appears unlikely to thrive in the plains of India; *E. elaeophora* does moderately well in Simla Hills, but has failed in the plains of N. India; *E. leucoxylon* grows in the Nilgiris and does moderately well in Simla Hills at 5,000 ft.; *E. macarthurii* has been tried without success in the plains of N. India; and *E. sideroxylon* has thrived in Kumaon Hills and at Abbottabad and Dehra Dun, although it has not done well in the plains. All these are trees (and not mallees) which do not appear to have attracted any serious attention in India from the point of view of the valuable essential oil, and only casual efforts have hitherto been made to cultivate them. If they have not been successfully cultivated at one place no attempts have been made to try them at other places. Nor has there been any serious attempt to augment their cultivation where they have thrived. So far as the essential oil is concerned, attention has only been paid to *E. globulus*, a species the exploitation of which has receded even in its own home.

Opinion has been divided in connection with the extension of cultivation of eucalypts in India. According to Troup (*loc. cit.*), some urge the great utility of these trees in supplying fast-growing timber and fuel, essential oils, tannins, and other products. Others are of the opinion that the extension of eucalyptus cultivation in India has gone far enough, that the trees are monotonous, and that in regions where they are capable of growing it is possible to grow indigenous species which yield better timber and are superior in every way except in so far as rapidity of growth is concerned. There is something to be said on both sides. It is true that as timber trees the eucalypts have not come up to expectation in India, even after a century of trial. On the other hand, where large supplies of quick-growing and, therefore, cheap fuel are required, it might be profitable to raise eucalyptus plantations provided the locality is favourable for its growth.

Troup concludes, "Provided, therefore, the extension of eucalyptus cultivation in India is restricted to cases where it is likely to be of distinct advantage, and does not involve the clearing of valuable indigenous



SL. No.	SPECIES	% OIL	SPECIFIC GRAVITY <sup>160</sup>	CONGEALING POINT	OPTICAL ROTATION	REFRACTIVE INDEX <sup>160</sup>	PRINCIPAL CONSTITUENTS	OTHER CONSTITUENTS (after Baker & Smith)	R/W COEFFICIENT
<b>Medicinal Oils</b>									
1	<i>E. polybractea</i>	1.5 to 2.5	0.9225 to 0.9267	-5° to -10°	+0.5° to +1.85°	1.4584 to 1.4613	Cineole 80 to 85%	Pinene, aromadendral*, alcoholic bodies, sesquiterpene	5
2	<i>E. australiana</i>	3 to 3.5	0.9172 to 0.920	-15° to -17°	+1° to +3.5°	1.4632 to 1.4644	Cineole 70 to 75% (phellandrene absent)	Terpineol, geraniol, pinene, piperitone, citral, paraffin	5
3	<i>E. dives</i> var. "C"	2 to 4	0.917 to 0.922	-12° to -16°	Up to +4°	1.4609 to 1.4640	Cineole 70 to 75%	α-Terpineol and citral	...
4	Mixture of <i>E. elaeophora</i> , <i>E. sideroxylon</i> , <i>E. leucoxydon</i>	0.8	0.9143 to 0.9224	-11° to -18°	+0.5° to +1°	1.4612 to 1.4624	Cineole 70 to 75% (phellandrene absent)	Pinene, sesquiterpene, small amounts of aldehydes, etc.	...
5	<i>E. encorifolia</i>	2	0.9152 to 0.9335	...	-1.35° to -3.8°	1.4654 to 1.4681	Cineole 70%	Pinene, aromadendral*, alcoholic bodies	7.5
6	<i>E. viridis</i>	1	0.926	-9°	+0.88°	1.4632	Cineole 70 to 80%	Pinene, aromadendral*, sesquiterpene	...
7	<i>E. dumosa</i>	1	0.9187	-18°	+5.5°	1.4620	Cineole 60 to 70%	Pinene and aromadendral*	...
8	<i>E. dives</i> (type)	2 to 4	0.8892 to 0.9063	...	-58° to -78°	1.4784 to 1.4806	<i>l</i> -Piperitone 40 to 50%, <i>l</i> -α-phellandrene 40%	Some alcoholic bodies, and sesquiterpene, cymene, and oranene	8
<b>Industrial Oils†</b>									
9	<i>E. phellandra</i>	3 to 4.5	0.902 to 0.905	...	-12° to -28°	1.4660 to 1.4695	<i>l</i> -α-Phellandrene, cineole 20 to 50%	Pinene, eudesmol, terpineol, geraniol	6
10	<i>E. dives</i> var. "A"	2 to 4	0.8630 to 0.8772	...	-44° to -66°	1.4759 to 1.4792	<i>l</i> -α-Phellandrene 60 to 80%, <i>l</i> -piperitone 5 to 15%	Piperitol and sesquiterpenes	...
11	<i>E. numerosa</i>	2.3 to 3	0.8904 to 0.8948	...	-25° to -35°	1.4755 to 1.4786	Mainly <i>l</i> -α-phellandrene	Piperitol, esters, sesquiterpene, piperitone	10
12	<i>E. numerosa</i> var. "A"	3 to 3.5	0.901 to 0.910	...	-46° to -65°	1.4790 to 1.4820	<i>l</i> -Piperitone 50%, <i>l</i> -α-phellandrene 40%	...	...
<b>Perfumery Oils</b>									
13	<i>E. macarthurii</i>	0.2 to 0.5 sometimes	0.9238 to 0.9292	...	+1.6° to +3.1°	1.4704 to 1.4744	Geranyl acetate 67 to 78%, geraniol 3 to 7%, eudesmol 15 to 17%	Pinene	1
14	<i>E. citriodora</i>	0.7 to 1.0	0.8658 to 0.8740	...	-0.2 to +1.5	1.4521 to 1.4563	Citronellal 70 to 85%	Esters and free alcohols	8

\* According to Penfold (*Private communication*, 1949), aromadendral has no separate existence, but, following Baker & Smith (1920, *Research on the Eucalypts and their Essential Oils*), it has been used throughout this chapter in a general sense to denote the presence of one or more of the three chemical constituents, namely cuminaldehyde, phellandral, and *l*-cryptone (formerly cryptal).

† Industrial use is chiefly in ore flotation.

timber species, there is much to be said in favour of it."

All this controversy is with regard to timber and fuel, and very little attention has hitherto been paid to the valuable essential oils derived from the eucalypts. In point of fact, nobody has ever considered the problem from this angle, although it must be reiterated that whatever quantity of oil is distilled from *E. globulus* in the Nilgiris is obtained as a by-product.

The above table gives average constants and other particulars about the commercial oils which are distilled in Australia (Penfold & Morrison, 1944, *Commercial Eucalyptus Oils*. *Bull. tech. Mus., Sydney*, No. 2: p. 1-36).

The following table gives average constants and other particulars of the oils obtainable from those eucalypts which have been introduced into India. The analytical figures are for the oils distilled in Australia from the Australian trees, as recorded by Baker & Smith in their monograph *Research on the Eucalypts and their Essential Oils*, 1920.

*Eucalyptus oil for use in medicine*—The British Pharmacopoeia (1932)\* requires that oil for use in medicine should contain not less than 70 per cent. w/w of cineole. It should be a colourless or pale-yellow liquid with an aromatic camphoraceous

\* We have not yet received a copy of the 1948 Pharmacopoeia, which has been published.



**PARTICULARS OF THE OILS OBTAINABLE FROM EUCALYPTS WHICH HAVE BEEN  
INTRODUCED INTO INDIA**

SL. No.	SPECIES	COMMON NAME	AVERAGE % OIL	SPECIFIC GRAVITY <sup>180</sup>	OPTICAL ROTATION	REFRACTIVE INDEX <sup>20°</sup>	SAPONIFICATION VAL.	PRINCIPAL CONSTITUENTS
1	<i>E. acmenioides</i>	White mahogany	0.09	0.9252	Not taken	1.5008	8.6	Pinene, phellandrene, sesquiterpene, cineole (traces)
2	<i>E. alpina</i>	...	0.36	0.8973	-2.8°	1.4756	2.6	Pinene, cineole, paraffin, sesquiterpene, polyterpenes
3	<i>E. amygdalina</i>	Black peppermint	1.80	0.8668 to 0.8848	-59.1° to -75.1°	1.4752 to 1.4781	2.9 to 3.2	Phellandrene, cineole, piperitone, sesquiterpene
4	<i>E. andrewsi</i>	New England peppermint	1.27	0.8646	-41.5°	1.4831	4.3	Phellandrene, piperitone, free alcohols, sesquiterpene, cineole (traces)
5	<i>E. bicolor</i>	Red box	0.85	0.9155	+5.5°	1.4675	8.4	Cineole, pinene
6	<i>E. bosistoana</i>	Ribbon box	0.97	0.9078	+9.26°	1.4675	3.2	Cineole, pinene, terpineol, sesquiterpene
7	<i>E. botryoides</i>	Bastard mahogany, bangalay	0.11	0.8774 to 0.8778	+23.75° (1st fraction)	1.4730	15.2 to 21.4	Pinene, sesquiterpene, esters, cineole (traces)
8	<i>E. calophylla</i>	Red gum	0.25	0.8756	+22.9°	1.4731	10.5	Pinene, cymene, sesquiterpene, esters, cineole (traces)
9	<i>E. capitellata</i>	Brown stringybark	0.11	0.9175	+4.4°	1.4771	4.3	Cineole, pinene, phellandrene, sesquiterpene
10	<i>E. cinerea</i>	Argyle apple	1.20	0.9113 to 0.9225	+2.5° to +4.1°	1.4649	14.4 to 24.0	Cineole (54%), pinene, esters, volatile aldehydes, sesquiterpene
11	<i>E. citriodora*</i>	Lemon-scented gum	0.70 to 1.0	0.8658 to 0.8740	-0.2° to +1.5°	1.4521 to 1.4563	...	Citronellal (70-85%), esters, free alcohols
12	<i>E. cladocalyx</i>	Sugar gum	0.10	0.8945	+8.1°	1.4779	7.7	Pinene, cineole, sesquiterpene
13	<i>E. coccifera</i>	...	0.61	0.8810	-35.8°	1.4840	4.0	Phellandrene, cineole, eudesmol, piperitone, sesquiterpene
14	<i>E. coriacea</i>	Scribbly gum, white gum	0.61	0.8947 to 0.8983	-27.0° to -20.3°	1.4845 to 1.4890	3.7 to 4.6	Phellandrene, pinene, piperitone, eudesmol, cineole, sesquiterpene
15	<i>E. cornuta</i>	Yate gum	1.20	0.9043	+10.1°	1.4601	25.4	Pinene, cineole (31%), amyl alcohol, esters, alcohols, sesquiterpene, volatile aldehydes
16	<i>E. corymbosa</i>	Bloodwood	0.06	0.883	-8.4°	1.4838	3.8	Pinene, aromadendral†, sesquiterpene, cineole
17	<i>E. crebra</i>	Narrow-leaved ironbark	0.16	0.8986	-10.8°	1.4787	6.2	Phellandrene, pinene, cineole, sesquiterpene
18	<i>E. dealbata</i>	White gum	0.86	0.9261	+3.8°	1.4648	2.1	Cineole (52%), pinene, sesquiterpene, liquid eudesmol
19	<i>E. deanei</i>	...	0.80	0.9216	-3.2°	1.4806	21.4	Pinene, cymene, aromadendral†, esters, sesquiterpene
20	<i>E. delegatensis</i>	White ash	1.70	0.8596 to 0.8676	-47.4° to -58.6°	1.4812 to 1.4863	3.1 to 3.5	Phellandrene, piperitone, cineole (traces)
21	<i>E. diversicolor</i>	Karri	0.80	0.8948 to 0.9145	+20.5° to +30.1°	1.4671 to 1.4748	41 to 53.2	Pinene, terpineol, geraniol and their esters, butyl butyrate, sesquiterpene, cineole (traces)
22	<i>E. dives*</i> (type?)	Broad-leaved peppermint	2 to 4	0.8892 to 0.9063	-58° to -78°	1.4784 to 1.4806	...	l- $\alpha$ -phellandrene (40%), l-piperitone (35-50%), alcoholic bodies, and sesquiterpene, and origanene
23	<i>E. elaeophora</i>	Bundy, half box	0.75	0.9191 to 0.9266	+3.1° to +5.7°	1.4663	8.7	Cineole (58%), pinene, eudesmol, aromadendral†, esters
24	<i>E. eugeniolides</i>	White stringybark	0.75	0.9122 to 0.9132	+3.4° to +4.8°	1.4694	6.9	Cineole (31%), pinene, sesquiterpene
25	<i>E. eximia</i>	White or yellow bloodwood	0.46	0.8998	+28.8°	1.4832	4.5	Pinene, sesquiterpene, liquid eudesmol, geraniol
26	<i>E. globulus</i>	Blue gum	0.92	0.913	+8.4°	1.4663	2.1	Cineole (57%), pinene, eudesmol, sesquiterpene, volatile aldehydes
27	<i>E. gomphocephala</i>	Tuart	0.03	0.8759	Not taken	1.4758	25.7	Phellandrene, pinene, esters
28	<i>E. goniocalyx</i>	Mountain gum	1.00	0.9117 to 0.9125	+4.35° to +7.2°	1.4689	13.3	Cineole (56%), pinene, eudesmol, sesquiterpene, esters, volatile aldehydes
29	<i>E. gunnii</i>	Cider gum, swamp gum	0.68	0.8984 to 0.9014	-4.3° to +4.7°	1.4743 to 1.4799	4.4 to 6.7	Pinene, phellandrene, cineole, liquid eudesmol, sesquiterpene
30	<i>E. haemastoma</i>	Scribbly gum, white gum	0.44	0.9065 to 0.9195	-5.5°	1.4902 to 1.4955	4.4 to 5.1	Phellandrene, cineole, eudesmol, geraniol, sesquiterpene

\* Data for this have been taken from Penfold & Morrison (1944. *Bull. tech. Mus., Sydney*, No. 2 : p. 1-36).

† According to Penfold (1949. *Private communication*), aromadendral has no separate existence, but, following Baker & Smith (1920. *Research on the Eucalypts and their Essential Oils*), it has been used throughout this chapter in a general sense to denote the presence of one or more of the three chemical constituents, namely cuminaldehyde, phellandral, and l-cryptone (formerly cryptal).

**PARTICULARS OF THE OILS OBTAINABLE FROM EUCALYPTS WHICH HAVE BEEN  
INTRODUCED INTO INDIA — (Continued)**

SL. No.	SPECIES	COMMON NAME	AVERAGE % OIL	SPECIFIC GRAVITY <sup>15°</sup>	OPTICAL ROTATION	REFRACTIVE INDEX <sup>20°</sup>	SAPONIFICATION VAL.	PRINCIPAL CONSTITUENTS
31	<i>E. hemiphloia</i>	Grey box, white box	0.58	0.9117	-6.85°	1.4853	5.8	Pinene, cineole, aromadendral†, cymene, alcoholic bodies, sesquiterpene
32	<i>E. leucoxydon</i>	Victorian iron-bark, white gum	0.78	0.8987	+9.2°	1.4711	3.8	Pinene, limonene, cineole, sesquiterpene
33	<i>E. linearis</i>	White peppermint	1.50	0.9036 to 0.9096	-9.9 to -23.1°	1.4677 to 1.4734	5.8	Phellandrene, cineole (46%) liquid eudesmol, sesquiterpene
34	<i>E. longifolia</i>	Woolly butt	0.54	0.9226	+2.86°	1.4681	3.6	Cineole (45%), pinene, sesquiterpene
35	<i>E. macarthurii*</i>	Paddy's river box	0.2 sometimes 0.5	0.9238 to 0.9292	+1.6° to +3.1°	1.4704 to 1.4744	...	Geranyl acetate (67-73%), geraniol (3-7%), eudesmol (15-17%)
36	<i>E. macrorhyncha</i>	Red stringybark, Victorian stringybark	0.29	0.929	-1.0° (1st fraction)	1.4745	8.4	Pinene, phella: trene, cineole, eudesmol, sesquiterpene
37	<i>E. maideni</i>	Blue gum	1.0	0.9247	+5.8°	1.4679	21.8	Cineole (50%), pinene, esters, sesquiterpene
38	<i>E. marginata</i>	Jarrah	0.22	0.8889 to 0.9117	-8.5° to -10.4°	1.4889	10.3 to 13.1	Pinene, cymene, cineole, aromadendral†, sesquiterpene, esters
39	<i>E. melanophloia</i>	Silver-leaved ironbark	0.11	0.8950	-23.5°	1.4893	11	Phellandrene, pinene, cymene, cineole, eudesmol, sesquiterpene
40	<i>E. melliodora</i>	Yellow box	0.87	0.9042 to 0.9321	+5.0° to +6.5°	1.4649	7.2 to 21.96	Cineole (52%), phellandrene, pinene, sesquiterpene, esters
41	<i>E. microcorys</i>	Tallow-wood	0.51	0.895	+18.3°	1.4690	10.6	Cineole, pinene, esters, sesquiterpene
42	<i>E. microtheca</i>	Coolabah, tangoon	0.48	0.8855	-27.2°	1.4838	4.3	Phellandrene, pinene, cineole, cymene, sesquiterpene
43	<i>E. obliqua</i>	Stringybark, messmate	0.70	0.8836 to 0.8902	-24.2° to -28.8°	1.4836 to 1.4877	7.2 to 8.1	Phellandrene, cineole, aromadendral†, cymene
44	<i>E. occidentalis</i>	...	0.95	0.9135	+9.0°	1.4717	2.5	Pinene, cineole (36%), aromadendral†, sesquiterpene
45	<i>E. paniculata</i>	White ironbark	0.10	0.901	+7.8°	1.4744	7.11	Pinene, cineole, alcoholic bodies, sesquiterpene
46	<i>E. patentinervis</i>	Half mahogany	0.26	0.8735	-15.2°	1.4891	6.2	Terpenes, limonene, citral, alcohols, sesquiterpene
47	<i>E. pilularis</i>	Blackbutt	0.11	0.885 to 0.903	-4.2° to +11.1°	1.4904	5.9	Phellandrene, pinene, cineole, sesquiterpene, liquid eudesmol
48	<i>E. piperita</i>	Sydney peppermint	0.80	0.9111	-2.7°	1.4781	11.0	Phellandrene, pinene, cineole, piperitone, eudesmol, sesquiterpene
49	<i>E. planchoniana</i>	Stringybark	0.02	0.9039	Not taken	Not taken	7.1	Phellandrene, sesquiterpene
50	<i>E. platypus</i>	...	0.82	0.9103	+10.2°	1.4639	15.2	Cineole (48%), pinene, esters, alcoholic bodies, sesquiterpene, volatile aldehydes
51	<i>E. polyanthemus</i>	Red box	0.83	0.9281	+5.4°	1.4679	20.7	Cineole (54%), pinene, esters, sesquiterpene
52	<i>E. populifolia</i>	Bimil box, poplar-leaved box	0.76	0.9207 to 0.923	+0.4° to +1.2°	1.4652	1.9	Cineole (62%), pinene, sesquiterpene
53	<i>E. propinqua</i>	Grey gum of the north coast, N.S.W.	0.24	0.8980	+4.4°	1.4731	8.4	Pinene, cineole (32%), aromadendral†, sesquiterpene
54	<i>E. pulverulenta</i>	...	2.22	0.9236	+2.12°	1.4629	18.4	Cineole (60%), pinene, esters, sesquiterpene
55	<i>E. punctata</i>	Grey gum	0.8	0.9160	+0.85°	1.4717	18.8	Cineole (55%), pinene, aromadendral†, esters, sesquiterpene
56	<i>E. redunca</i>	White gum	1.2	0.9097	+13.5°	1.4663	2.4	Cineole (38%), pinene, sesquiterpene
57	<i>E. regnans</i>	Giant gum	0.78 to 0.83	0.8802 to 0.8879	-24.4° to -31.1°	1.4882 to 1.4901	13.2 to 15.4	Phellandrene, eudesmol, geraniol, esters, piperitone, sesquiterpene
58	<i>E. resinifera</i>	Australian red mahogany	0.42	0.9098	+2.25°	1.4698	9.2	Cineole (50%), pinene, sesquiterpene, esters
59	<i>E. robusta</i>	Swamp mahogany	0.16	0.8777	+4.0°	1.4744	9.1	Pinene, sesquiterpene, cineole, esters
60	<i>E. rostrata</i>	Murray red gum	0.27	0.8955 to 0.9047	-11.8° to -14.5°	1.4839 to 1.4890	6.1 to 10.6	Cineole, pinene, phellandrene, cymene, aromadendral†
61	<i>E. rubida</i>	Candlebark	0.01 to 0.11	0.9067 to 0.9209	-1.1° to +4.4°	1.4892	3.2 to 12.3	Pinene, cineole, phellandrene, esters, sesquiterpene
62	<i>E. rudis</i>	Flooded gum, swamp gum	1.2	0.9068	+10.1°	1.4695	3.2	Pinene, cineole (38%), sesquiterpene, volatile aldehydes

\* See footnote on p. 210.

† See footnote on p. 210.

**PARTICULARS OF THE OILS OBTAINABLE FROM EUCALYPTS WHICH HAVE BEEN  
INTRODUCED INTO INDIA — (Continued)**

SL. No.	SPECIES	COMMON NAME	AVERAGE % OIL	SPECIFIC GRAVITY <sup>15°</sup>	OPTICAL ROTATION	REFRACTIVE INDEX <sup>20°</sup>	SAPONIFICATION VAL.	PRINCIPAL CONSTITUENTS
63	<i>E. saligna</i>	Sydney blue gum	0.12	0.8731 to 0.8846	+1.1°	1.4789 to 1.4795	26.2 to 35.5	Pinene, cymene, esters, sesquiterpene, cineole (traces)
64	<i>E. salmonophloia</i>	Salmon-coloured gum	1.44	0.9076	+6.3°	1.4681	4.9	Pinene, cineole (48%), aromadendral†, sesquiterpene
65	<i>E. salubris</i>	Gimlet gum	1.39	0.902	-5.8°	1.4784	18.9	Pinene, cymene, aromadendral†, esters, cineole, sesquiterpene
66	<i>E. siderophloia</i>	Sydney ironbark	0.06	0.9067	+14.5°	1.4943	4	Pinene, phellandrene, alcoholic bodies, sesquiterpene, cineole (traces)
67	<i>E. sideroxylon</i>	Red flowering iron-bark	0.60	0.9189 to 0.9227	+1.4° to +3.2°	1.4668	2.5	Cineole (60%), pinene, sesquiterpene
68	<i>E. sieberiana</i>	Mountain ash	0.5	0.8880	-43.3°	1.4820	2.5	Phellandrene, piperitone, cineole, cymene, sesquiterpene
69	<i>E. smithii</i>	Gully ash, white ironbark, white-top	1.80	0.9133 to 0.9192	+4.7° to +6.4°	1.4616 to 1.4672	2.4	Cineole (73-85%), pinene, eudesmol, paraffin
70	<i>E. stellulata</i>	Black sally, lead gum	0.3	0.871	-22.7°	1.4845	2.1	Phellandrene, cineole, sesquiterpene
71	<i>E. stricta</i>	...	0.5	0.9246	-0.35°	1.4654	2.5	Cineole (58%), pinene, eudesmol, aromadendral†
72	<i>E. Stuartiana</i>	Apple box	0.4	0.916	+4.8°	1.4652	14.2	Cineole (53%), pinene, esters, sesquiterpene
73	<i>E. leucicornis</i>	Forest red gum	0.5	0.9158 to 0.9128	-9.4° to -11.8°	1.4877 to 1.4906	26.7	Pinene, cymene, cineole, aromadendral†, sesquiterpene
74	<i>E. trachyphloia</i>	Bloodwood	0.2	0.8873 to 0.8929	+8.4° to +9.8°	1.4844	3.2	Pinene, sesquiterpene, aromadendral†
75	<i>E. umbra</i>	Stringybark, white mahogany	0.6	0.8970	+18.7°	1.4639	7.1	Pinene, cineole, sesquiterpene
76	<i>E. urnigera</i>	Urn gum	1.13	0.9088	+11.8°	1.4652	18.3	Cineole (53%), pinene, esters, volatile aldehydes, sesquiterpene
77	<i>E. viminalis</i>	Manna gum	0.55	0.9044 to 0.9162	+3.7° to +4.2°	1.4707 to 1.4798	6.1 to 9.5	Pinene, phellandrene, cineole, sesquiterpene
78	<i>E. virgata</i>	...	0.54	0.8883 to 0.9154	-20.9°	1.4810 to 1.4958	3.3 to 5.7	Phellandrene, eudesmol, cineole (traces), piperitone, sesquiterpene

† See footnote on p. 210.

odour, pungent and camphoraceous taste followed by a sensation of cold, soluble in 5 volumes of alcohol (70 per cent.), and should have sp. gr. (15.5°/15.5°) 0.910 to 0.930,  $[\alpha]$  -5° to +5°, refractive index at 20° 1.458 to 1.470. When 1 ml. of oil is mixed with 2 ml. of glacial acetic acid and 5 ml. of petroleum spirit (b.p. 50° to 60°) and 2 ml. of saturated aqueous solution of sodium nitrite are added and the mixture gently shaken, the nonformation of a crystalline precipitate in the upper layer is the indication of the absence of much phellandrene. The pharmacopoeia also lays down the limit of aldehydes.

The Pharmacopoeia of the U.S.A. (1942) also requires that the eucalyptus oil for use in medicine should contain not less than 70 per cent. of cineole (eucalyptol).

Formerly the oil from *E. globulus* was highly prized for use in medicine, and took the first place. The B.P. requirements in 1914 were not less than 55 per cent. of cineole, the constituent for which the oil is valued in medicine, but this has since been

raised to not less than 70 per cent. This change has resulted in getting the oil from those species in which the oil is richer in cineole and larger in amount. Whereas, the leaves and twigs of *E. globulus* furnish less than 1 per cent. of oil (containing about 60 per cent. cineole) those from *E. polybractea* yield double and from *E. australiana* four times the amount (containing 70 to 85 per cent. of cineole). None of these latter species have ever been tried in India, despite the fact that oil from the Indian-grown *E. globulus* does not conform to B.P. standard, not only with regard to cineole content but also in other respects.

The essential oil from *E. dives* (type) is rich in piperitone content. Piperitone is easily convertible into menthol and thymol, which are used in medicine. This species has hardly received any attention in India, although the demand for thymol and menthol is insatiable. Australia supplies India with large quantities of thymol.

The popularity of disinfectants and deodorants based upon eucalyptus oils has

greatly stimulated production in Australia of those grades of oils which are rich in phellandrene and contain 25 to 30 per cent. of cineole. This aspect of the utility of eucalyptus oils has received no attention in this country.

*Eucalyptus oils for use in perfumery*—Eucalyptus oils from two species find use in perfumery. These are *E. citriodora* oil which is rich in citronellal content and *E. macarthurii* oil which has a high content of geranyl acetate. These species also have hardly received any attention in India.

*Eucalyptus oils for use in industry*—Oils from some species, such as *E. dives* var. "A" and *E. phellandra*, are used in special processes, such as the "flotation" process for ore concentration. These again have received little attention in India.

*Leaf material for eucalyptus oil*—The suitability of a eucalyptus as a commercial source of oil depends upon the composition and yield of the oil; also upon the accessibility of the species and the yield of the leaves. Many species, such as *E. globulus*, *E. citriodora* and *E. macarthurii*, although useful for the production of oil, grow to large dimensions. Leaves and terminal branches are the only parts from which the oil is distilled, and to lop the branches from such tall and none too strong trees is both expensive and dangerous. The felling of the trees is, therefore, usually favoured in Australia by experienced distillers, although it may appear extravagant. By cutting the tree within a foot or two of the ground, a strongly foliaceous "sucker growth" springs up soon, and this may be cut again and again as required for distillation. With many eucalypts coppice is readily and abundantly produced, and the leaf material from such shrubby regrowth is more easily gathered. The vitality shown by the eucalypts is remarkable, and repeated cutting does not destroy the reproductive capacity of the trees.

Eucalypts, however, do not all grow to a large size, the mallees more particularly. This shrubby growth is more useful for oil distillation than the taller species, besides the fact that several mallees are rich in oil content, and the oil rich in cineole. In mallees several stems spring from an expanded rootstock, and these form a bushy growth which is very convenient for harvesting. Three mallees particularly have attracted great attention in Australia, on account of the high yield of cineole-rich oils. These are

*E. polybractea* (blue mallee) of New South Wales, *E. cneorifolia* (narrow-leaved mallee) of the Kangaroo Island and *E. odorata* Behr. (box tree or peppermint) of New South Wales, Victoria, and South Australia. The bulk of South Australian oil is distilled from these species (Read, 1944. *Endeavour*, 3: 47). Another mallee yielding somewhat lesser amount of cineole-rich oil is *E. dumosa* (bull mallee), which is also exploited to an extent. *E. polybractea* is, however, the best-known and most exploited of these mallees.

In order to secure an abundance of new leaves from the mallees, particularly *E. polybractea*, the distiller in Australia employs a heavy roller to crush down the whole of the natural growth of the mallee scrub. When the crushed material has sufficiently dried, it is burnt down. In a few weeks the irrepressible eucalypt reappears and the landscape is covered by young growth. In 12 to 18 months the thick growth of the mallee is ready for harvesting for distillation.

Not only are the mallees remarkable for their vitality and rapid growth, but a number of arboreal eucalypts are also notable for their fast growth. For example, *E. macarthurii*, when grown from a seed, can be harvested in 20 to 24 months and the oil distilled from its leaves contains 70 per cent. of geranyl acetate—a constituent valuable in perfumery. Another example of a quick-growing tree is *E. smithii*, whose leaf oil contains 73 to 85 per cent. of cineole. This has been found to grow to a height of 22 ft. in three years, and can be repeatedly harvested for oil distillation. It is possible to trim it to a bush when the collection of leaves is made easy, besides obtaining an abundance of foliage. It has been suggested that *E. globulus*, which has been cultivated in India, might behave in this manner.

*Variation in character and yield of oil due to age*—In Australia it has been observed with some eucalypts that the oil from the young and old leaves do not differ much in their constituents. In some cases the young leaves yield a higher percentage of oils, but lower amounts of the constituents for which they are prized. In India, it is said that distillers prefer leaves from *E. globulus* which is 16 to 17 years old, although no data are available to uphold this view. In any case, whatever species is cultivated in India, it is advisable to investigate both the yield and the constitution of oil according

to the age of the eucalypt. If the leaves from younger trees give a higher yield of the oil, it may still be rich enough in the constituent of the oil for which it is prized to justify the exploitation of younger trees. It is only after such investigations that the best manner and time of collection for each species can be worked out economically.

*Distillation of eucalyptus oil*—Eucalyptus oil can be distilled from the leaves and terminal branchlets by steam at ordinary pressure, but preferably at 10 lbs. steam pressure. Direct-fired stills can be used, but the oil obtained is crude and requires rectification. For this reason, wherever possible, steam distillation should be resorted to. Since eucalyptus oil is so easily distilled, and the leaf material not difficult to collect, it is only natural that very crude apparatus is often employed, although in modern distilleries steam-distilling plants are in operation. The erection of a permanent distillery for the purpose of course depends upon the availability of leaves and abundance of water. In general, it is advantageous to have distilling plants of small size, which are capable of being dismantled and taken from place to place in the forest areas.

The leaves of all eucalyptus trees are evergreen so that distillation can go on all the year round except that the oil in winter is low. It has, however, been demonstrated that a particular species of *Eucalyptus* will produce, from average material, an oil practically constant in composition and general characters and also gives a fairly uniform yield, provided the material is well selected and does not possess too many branchlets. This factor is of great help in laying down the standards and specifications, and building up trade in these oils.

The process of distilling eucalyptus oil in India has been described by Natarajan (1929. *Industr. Engng. Chem. News Edition*, May 10; *vide Perfum. essent. Oil Rec.*, 1929, 20:192). He describes the process as under:—

The manufacture of eucalyptus oil from *E. globulus* leaves is carried on extensively in the Nilgiri district. Eucalyptus trees are grown largely on the hills, as they are used for fuel and regularly cut for this purpose. There are about 100 distilleries in the district. The oil is used for medicinal purposes in hospitals and also for removing incrustations in boilers and locomotives. It is exported in large quantities to N. India and elsewhere.

The still used for the manufacture of eucalyptus oil is ordinarily 4 ft. high and 2½ ft. in diameter, made of copper plates over which the leaves are placed so that they will not come in direct contact with the heat. Leaves of mature trees, 16 to 17 years old, are preferred to leaves of younger trees. They are dried for three days after picking before being distilled. Leaves dried in the shade give a greater yield of oil. Before the heating commences, the still is filled with water and the required quantity of leaves is placed over the perforated plate. The cover of the still is tightly closed and the vapour is allowed to escape through a pipe into a tub, after first passing through a column of cold water. The condensed vapour contains a large percentage of water. The oil, being lighter, rises to the surface and the water is allowed to pass out through a tap at the bottom of the vessel. Generally boiling is continued for 8 hours, the water in the still being maintained to the same level by constant addition as it evaporates. The crude oil thus collected is refined by mixing it with one sixth the quantity of water and adding a small amount of caustic soda. The mixture is again distilled in a similar but a smaller still and the oil is passed through filter papers until it is rid of all impurities.

The average capacity of a still is to hold 800 lbs. of leaves and 36 gals. of water. The yield from 800 lbs. of leaves is 96 ozs. of refined oil. This works out to a yield of 0.75 per cent. of oil.

The following is the description of the distillation of eucalyptus oil in Australia given by Baker and Smith (1920. *A Research on the Eucalypts and their Essential Oils*: p. 446):—

"In the smaller distilling plants, in New South Wales particularly, it has become customary to generate the steam in the vessel in which the leaves are packed. A very general method is to employ one or more of the common square 400-gallon iron tanks, to insert a grating of some kind at about 8 inches from the bottom to support the leaves, and to fill the space below with water. The tanks are placed upon a suitable foundation and the fires lighted below. When the water boils the steam passes through the leaves, carrying the oil with it. The condenser and still attachments take various forms, but all are constructed with some regard to main principles of condensation. In the more simply constructed

plants, particularly in localities where water is abundant, the condensation is brought about by laying a sufficient length of pipe from the still, in a running stream if possible, bending the end of the pipe outwards through some protection, and collecting the condensed water and oil in a receiver in which the oil separates, and is drawn off either automatically or otherwise.

"In simple distilling plants of this nature no provision is usually made for unloading the spent leaves by mechanical means, this operation being performed by manual labour, so that, with a still of this description, it is evident that a minimum of result is obtained with a maximum of effort; but this primitive method of distillation will continue to be employed because the plant is cheap, easily dismantled for removal, and can be worked by one or two men, by families, or by small communities.

"The lid attachments to these tank stills are made in various ways, the most common being to remove the top, and rivet a right-angled flange around the outside. The lid is a flat sheet of iron, and between this and the flat surface of the flange the joint is made, by inserting packing of some sort, either with clay or without, and fastening the whole with stout steel clips. Another method is to have the flange turned inwards and to fasten stirrups on the outside of the tank. The lid is a flat sheet of iron in this case also, and the joint made steam-tight in the usual way and fastened tightly by the aid of wedges.

"It was considered at one time that the correct arrangement for a Eucalyptus oil still must include a copper still-head, to allow of easy exit of the steam from the tank to the condenser, but in the majority of cases this copper outlet is now discarded, as it was found to be unnecessary and costly. It must be remembered, too, that in nearly every case Eucalyptus oils contain free acid, and this, when present in sufficient amount, attacks the copper, the dissolved portion being conveyed into the oil, giving it a green colour. How injurious this may be is seen from the results we have recorded under *E. cinerea*. The outlet for the steam is now made in a simple manner by inserting a 2 or 3 inch iron pipe through the side of the tank just below the top. No difficulty is experienced, and practically all the oil is obtained from the leaves in this way."

The attention of the reader is also invited to an excellent bulletin entitled

*Guide to the extraction of eucalyptus oil in the field* by Penfold & Morrison (1945. *Bull. tech. Mus.*, Sydney, No. 4: p. 1-24).

*E. globulus* oil produced in the Nilgiris—Puran Singh (1917. *Indian For. Rec.*, 5: 301) states that the shade-dried leaves containing 10 per cent. moisture give at least 50 per cent. greater yield of oil than the same quantity of green leaves containing 41 per cent. moisture. He also recommends that the leaves should not be exposed directly to the sun during drying but that they should be dried in shade. The leaves yield 0.8 to 1.2 per cent. (Ramaswamy, Rao & Guha, 1946. *J. Indian Inst. Sci.*, 28A: 57), although Puran Singh obtained even 1.5 per cent. of the oil from mature tree of original growth. Puran Singh also states that the leaves from one year coppice shoots contain more oil than those from 10 years coppice growth, bulk for bulk. The average yield from the leaves in Australia has been recorded as 0.92 per cent. (Baker & Smith, *loc. cit.*).

Below are given the particulars about the Nilgiri oil as compared to the Australian oil:—

	AUSTRALIAN OIL	NILGIRI OIL (after Puran Singh)	NILGIRI OIL (after Ramaswamy <i>et al.</i> )
Specific gravity	0.913	0.9065 to 0.9155	0.902
Optical rotation	+8.4°	+5.27° to +9.39°	+9.68°
Refractive index	1.4663	1.463 to 1.466	1.4608
Acid val.	...	0.18 to 1.04	6.3
Saponification val.	2.1	8.90 to 20.0	37.5
Saponification val. after acetylation	...	17.0 to 21.68	92.2
Cineole	57%	54 to 60%	62%
Solubility in alcohol	1.5 in 70%	1 in 80%	Insoluble in 10 vol. 70% alcohol

Ramaswamy *et al.* also give the following additional constituents:—

Lower boiling alcohols, aldehydes, ketones and acids 2.5 per cent., pinenes 24 per cent., pinocarveol 0.4 per cent., cuminaldehyde 0.5 per cent., sesquiterpene alcohols 5.0 per cent., aromadendrene 1.0 per cent., phellandrene nil, phenol 0.3 per cent., and residue 3.0 per cent.

It will be seen that cineole content of the Nilgiri oil is well below the B.P. standard. Optical rotation and solubility in alcohol also differ from the limits laid down by B.P. Aldehydes which were not found by Puran Singh have been reported from the Nilgiri oil. Unless, therefore, the oil can be properly rectified, this would not come up to the B.P.

standard. Nonetheless, it is produced in India to a large extent and used in medicine. In Australia, however, this has now gone out of market having been replaced by cineole-rich oils from such species as *E. polybractea* which not only yield greater amounts of oil from the leaves but the oil itself is richer in cineole content than that from *E. globulus*.

Writing about the Indian eucalyptus oil, Chopra (1933. *Indigenous Drugs of India*: p. 170) says, "In the field of medicine, the Indian oil should have better prospects. Phellandrene, which is present in the Australian oil to a fairly large extent, is very irritant to the bronchial mucosa, especially if inhaled and has been considered to be powerfully depressant to the heart. The British Pharmacopoeia tests expressly exclude oils containing much of this principle. The butyric and valerianic aldehydes also are obnoxious constituents in the Australian oil. Both these constituents are absent from the Indian oil and therefore this should merit better consideration by physicians as this oil is less likely to produce coughing and other unpleasant side-effects." Obviously these remarks were made in the light of 1914 British Pharmacopoeia which required a minimum of 55 per cent. of cineole, as against the minimum of 70 per cent. in the 1932 edition, which has also laid down different specifications for optical rotation and has specified the refractive index and also expressly laid down a limit of aldehydes.

McKern & Morrison (1947. *Aust. J. Pharm.*, 28: 858, 861; *vide Chem. Abstr.*, 1948, 42: 6007) have reviewed the literature on the therapeutic effects of phellandrene, and conclude that phellandrene is not more toxic than other terpenes in eucalyptus oil. According to them, its exclusion by the British Pharmacopoeia is unjustified.

*Constituents of eucalyptus oil*—Altogether about 40 distinct chemical constituents have been isolated or detected from oils belonging to this genus. Most of these are hydrocarbons or oxygenated derivatives belonging to the terpene series, such as cineole, citral, citronellal, eudesmol, geraniol, limonene, phellandrene, pinene, and piperitone; in addition there are aliphatic and aromatic constituents. A list of these is given below:—

*Acids*—Acetic and formic.

*Alcohols*—Geraniol, terpineol, piperitol, globulol, pinocarveol, eudesmol, methyl

alcohol, ethyl alcohol, butyl alcohol, isobutyl alcohol, and isoamyl alcohol.

*Aldehydes*—Cuminal, phellandral, cryptal,\* citral, citronellal, butyraldehyde, and valeraldehyde.

*Alkyl benzene*—Cymene.

*Esters*—Geranyl acetate, butyl butyrate, amyl eudesmate, valeric ester, terpinyl acetate, and terpinyl butyrate.

*Ketone*—Piperitone.

*Paraffin*—Solid hydrocarbon (deposits in oils).

*Phenols*—"Tasmanol" and *p*-isopropylphenol (formerly australol).

*Sesquiterpene*—Aromadendrene.

*Terpenes*—Pinene, phellandrene, limonene, dipentene, and  $\gamma$ -terpinene.

According to Penfold & Morrison (1944. *Commercial Eucalyptus Oils. Bull. tech. Mus., Sydney*, No. 2: p. 1-36), the following are the uses of the principal constituents of eucalyptus oils:—

"1. *Volatile aldehydes* (principally isovaleric), are used in various proprietary preparations including disinfectants, sheep dips, etc.

"2. *Pinene* and other terpenes are used in the manufacture of certain blended turpentine.

"3. *Cineol* [e], the principal constituent of medicinal eucalyptus oils, has a very restricted use. Production rarely exceeds a few tons per annum. It is used in pharmacy and confectionery and to a limited extent as a clothes cleaner.

"4. *Phellandrene* is obtained as a by-product in the separation of piperitone from *Eucalyptus dives* and allied species, and is in heavy demand as a general solvent. In association with piperitone and other constituents it is sold as a blended eucalyptus oil for mineral flotation.

"5. *Terpineol*—the well-known perfumery constituent of hyacinth odour—is separated from the high boiling fractions of *Eucalyptus Australiana*, *E. dives* var. 'C', etc.

"6. *Eudesmol*, the principal sesquiterpene alcohol of eucalyptus oils, is a very satisfactory fixative for perfumes required for industrial purposes.

"7. *Eudesmyl acetate*. This is prepared by acetylating eudesmol. It is used as a

\* This is now known as *l*-cryptone (Penfold, 1949. *Private communication*).

war-time substitute for oil for Bergamot and its constituents. It blends fairly well with lavender oil.

- "8. *Piperitone* (commercial), assaying 90% to 95% ketone. This ketone is prepared by a well-known Victorian firm. Piperitone of high laevo-rotation is available in ton lots. It is the raw material for the manufacture of synthetic thymol and menthol."

The Australian eucalypts have been grouped according to the constituents they possess.

For example, Group I consists of:—

*E. acaciaeformis* Deane & Maiden, *E. alpina* Lindl., *E. botryoides* Sm., *E. calophylla* R. Br., *E. carnea* R. T. Baker, *E. corymbosa* Sm., *E. dextropinea* R. T. Baker, *E. eximia* Schau., *E. intermedia* R. T. Baker, *E. laevopinea* R. T. Baker, *E. nigra* R. T. Baker, *E. nova-anglica* Deane & Maiden, *E. phlebophylla* F. Muell. ex Miq., *E. robusta* Sm., *E. rydalsensis* R. T. Baker & H. G. Smith, *E. saligna* Sm., *E. terminalis* F. Muell., *E. tessellaris* F. Muell., and *E. trachyphloia* F. Muell. All of these yield an oil consisting largely of pinene, without phellandrene. Cineole is almost or quite absent.

Group II consists of the following which yield an oil in which pinene and cineole are predominant, cineole not exceeding 40 per cent. Phellandrene and aromadendral\* are absent:—

*E. bauerleni* F. Muell.†, *E. blaxlandi* Maiden & Cambage, *E. bosistoana* F. Muell., *E. cladocalyx* F. Muell., *E. conica* Deane & Maiden, *E. cornuta* Labill., *E. eugenioides* Sieb. ex Spreng., *E. fasciculosa* F. Muell., *E. hemilampra* F. Muell., *E. intertexta* R. T. Baker, *E. lactea* R. T. Baker, *E. lehmanni* Preiss ex Schau., *E. leucoxydon* F. Muell., *E. maculata* Hook., *E. megacarpa* F. Muell., *E. microcorys* F. Muell., *E. paludosa* R. T. Baker, *E. paniculata* Sm., *E. quadrangulata* Deane & Maiden,

\* According to Penfold (1949. *Private communication*), aromadendral has no separate existence, but, following Baker & Smith (1920. *Research on the Eucalypts and their Essential Oils*), it has been used throughout this chapter in a general sense to denote the presence of one or more of the three chemical constituents, namely, cuminaldehyde, phellandral, and *l*-cryptone (formerly cryptal).

† There exists considerable confusion regarding the spelling of *E. bauerleni*. It has been variously spelt as *E. baeuerleni*, *E. baecuerlenii*, *E. bauerlenii*, and *E. bauerlesii*. All these spellings are incorrect. F. Mueller, in the *Victorian Naturalist*, 1890: p. 76, spelt it as *E. bauerleni*, which, of course, must stand. The version in *Index Kewensis*, viz., *E. bauerlesii*, is evidently a misprint.

*E. redunca* Schau., *E. rudis* Endl., *E. santaliifolia* F. Muell., *E. umbra* R. T. Baker, *E. viminalis* Labill., var. "A", and *E. wilkinsoniana* R. T. Baker.

In Group III class (a) are placed the following which yield an oil consisting principally of cineole and pinene, in which cineole exceeds 40 per cent. but is under 55 per cent. Phellandrene is absent and aromadendral\* very rarely occurs:—

*E. accedens* Fitzg., *E. behriana* F. Muell., *E. bicolor* A. Cunn. ex Hook., *E. calycogona* Turcz., *E. camphora* R. T. Baker, *E. cinerea* F. Muell. ex Benth., *E. dalympleana* Maiden, *E. dealbata* A. Cunn. ex. Schau., *E. gullicki* R. T. Baker & H. G. Smith‡, *E. longifolia* Link & Otto, *E. maideni* F. Muell., *E. platypus* Hook., *E. polyanthemus* Schau., *E. resinifera* Sm., *E. rodwayi* R. T. Baker & H. G. Smith, *E. rossii* R. T. Baker & H. G. Smith‡, *E. rostrata* Schlecht., var. *borcalis* R. T. Baker & H. G. Smith, *E. salmonophloia* F. Muell., *E. secana* Maiden, *E. stuartiana* F. Muell. ex Miq., *E. unialata* R. T. Baker & H. G. Smith, *E. urnigera* Hook. f., and *E. vernicosa* Hook. f.

In Group III class (b) are placed the following. In these the oil consists principally of cineole and pinene but, unlike in Group II and Group III (a), cineole exceeds 55 per cent. Phellandrene and aromadendral are absent:—

*E. australiana* R. T. Baker & H. G. Smith, *E. bridgesiana* R. T. Baker, *E. cordata* Labill., *E. costata* R. Br. ex F. Muell., *E. globulus* Labill., *E. goniocalyx* F. Muell., *E. longicornis* F. Muell., *E. maculosa* R. T. Baker, *E. moorei* Maiden & Cambage, *E. morrisii* R. T. Baker, *E. muelleri* J. B. Moore, *E. nepeanensis* R. T. Baker & H. G. Smith§, *E. parramattensis* C. Hall, *E. parvifolia* Cambage,

\* See first footnote in the previous column.

† *Eucalyptus gullicki* was described by R. T. Baker & H. G. Smith in *Research on Eucalypts and their Essential Oils*, 1920: p. 128, but it has not been included in *Index Kewensis*. The Kew authorities, whose attention has been drawn to this omission, inform us that it will now be included in the eleventh Supplement.

‡ The original spelling as given by R. T. Baker & H. G. Smith in *Research on Eucalypts and their Essential Oils*, 1902, is *Eucalyptus rossii*. The spelling given in *Index Kewensis*, viz., *E. rossei*, is evidently a misprint.

§ *Eucalyptus nepeanensis* was described by R. T. Baker & H. G. Smith in *Research on Eucalypts and their Essential Oils*, 1920: p. 167, but it has not been included in *Index Kewensis*. The Kew authorities, whose attention has been drawn to this omission, inform us that it will now be included in the eleventh Supplement.



*E. perriniana* F. Muell., *E. populifolia* Hook., *E. pulverulenta* Sims, *E. pumila* Cambage, *E. sideroxylon* A. Cunn., *E. smithii* R. T. Baker, and *E. squamosa* Deane & Maiden.

Group IV class (a) consists of the following. In this the oil has over 40 per cent. cineole, but pinene diminishes and aromadendral makes appearance, thus approaching the typical "boxes". Phellandrene is absent :—

*E. cneorifolia* DC., *E. cosmophylla* F. Muell., *E. dumosa* A. Cunn. ex Schau., *E. elaeophora* F. Muell., *E. odorata* Behr., *E. oleosa* F. Muell., *E. polybractea* R. T. Baker, *E. punctata* DC., *E. stricta* Sieb. ex Spreng., and *E. tereticornis* Sm., var. *cineolifera* R. T. Baker & H. G. Smith.

Group IV class (b) consists of the following. In this the oil contains over 40 per cent. of cineole, but phellandrene is making its appearance, thus approaching the more pronounced phellandrene-bearing oils :—

*E. considiniana* Maiden\*, *E. linearis* Dehnh., *E. melliodora* A. Cunn. ex R. T. Baker & H. G. Smith, *E. ovalifolia* R. T. Baker, var. *lanceolata* R. T. Baker & H. G. Smith, and *E. risdoni* Hook. f.

In Group V are placed the following. In this the oil consists largely of cineole, pinene and aromadendral, but cineole does not exceed 40 per cent. Phellandrene is usually absent :—

*E. affinis* Deane & Maiden, *E. albens* Miq., *E. deanei* Maiden, *E. exserta* F. Muell., *E. fletcheri* R. T. Baker, *E. gracilis* F. Muell., *E. hemiphloia* F. Muell., *E. marginata* Sm., *E. occidentalis* Endl., *E. propinqua* Deane & Maiden, *E. punctata* DC., var. *didyma* R. T. Baker & H. G. Smith, *E. rostrata* Schlecht., *E. rudderi* Maiden, *E. salubris* F. Muell., *E. tereticornis* Sm., *E. uncinata* Turcz., *E. viridis* R. T. Baker, and *E. woollsiana* R. T. Baker.

In Group VI are included the following. In this the oil consists principally of pinene, cineole, and phellandrene, but the cineole does not exceed 40 per cent. :—

*E. acervula* Hook. f., *E. acmenioides* Schau., *E. angophoroides* R. T. Baker, *E. bancrofti*

Maiden\*, *E. caerulea* R. T. Baker & H. G. Smith\*, *E. capitellata* Sm., *E. crebra* F. Muell., *E. dawsoni* R. T. Baker, *E. fastigata* Deane & Maiden, *E. fraxinoides* Deane & Maiden, *E. gunnii* Hook. f., *E. haemastoma* Sm., *E. irbyi* R. T. Baker & H. G. Smith, *E. laseroni* R. T. Baker, *E. macrorhyncha* F. Muell., *E. melanophloia* F. Muell., *E. microtheca* F. Muell., *E. obliqua* L'Hérit., *E. ovalifolia* R. T. Baker, *E. pilularis* Sm., *E. planchoniana* F. Muell., *E. rubida* Deane & Maiden, *E. siderophloia* Benth., and *E. viminalis* Labill.

Group VII class (a) consists of the following. The oil contains largely phellandrene, cineole and piperitone, but cineole does not exceed 40 per cent. :—

*E. amygdalina* Labill., *E. amygdalina* Labill. var. *nitida* Benth., *E. coccifera* Hook. f., *E. luehmanniana* F. Muell., *E. phellandra* R. T. Baker & H. G. Smith, *E. piperita* Sm., and *E. vitrea* R. T. Baker.

Group VII class (b) consists of the following. In this the oil contains largely phellandrene and piperitone, but cineole is almost, if not quite, absent :—

*E. andrewsi* Maiden, *E. campanulata* R. T. Baker & H. G. Smith, *E. coriacea* A. Cunn. ex Schau., *E. delegatensis* R. T. Baker, *E. dives* Schau., *E. gomphocephala* DC., *E. oreades* R. T. Baker, *E. radiata* Sieb. ex DC., *E. regnans* F. Muell., *E. sieberiana* F. Muell., and *E. taeniola* R. T. Baker & H. G. Smith.

Group VIII consists of the following. Cineole is almost or quite absent in the oil :—

*E. aggregata* Deane & Maiden, *E. apiculata* R. T. Baker & H. G. Smith, *E. citriodora* Hook., *E. ligustrina* DC., *E. macarthuri* Deane & Maiden, *E. marsdeni* C. Hall, *E. patentinervis* R. T. Baker, *E. staigeriana* F. Muell. ex Bailey, *E. stellulata* Sieb. ex DC., and *E. virgata* Sieb. ex DC.

*Production of eucalyptus oil in Australia*—According to Penfold & Morrison (1944. Commercial Eucalyptus Oils. Bull. tech. Mus., Sydney, No. 2 : p. 1-36), the total annual production of eucalyptus oil in Australia during the years 1941 and 1942 was about 230,000 gallons (120,000 gallons of industrial oils and 110,000 gallons of medicinal oils). The export of the oils increased from about

\* The original spelling by Maiden in *Proc. linn. Soc. N.S.W.*, 1904, 29 : 475 is *Eucalyptus considiniana* and this is followed in *Index Kewensis*. But this must be regarded as a faulty formation since it was named after First-Assistant-Surgeon D. Considen (not Considine). The alteration by Baker & Smith in *Research on Eucalypts and their Essential Oils*, 1920 : p. 200 to *E. considiniana* is, therefore, justified, and should be followed.

\* *Eucalyptus bancrofti* was described by Maiden in *Crit. Rev. Gen. Euc.*, 1917, 4 : 14, and *E. caerulea* by R. T. Baker & H. G. Smith in *Research on Eucalypts and their Essential Oils*, 1920 : p. 271, but these have not been included in *Index Kewensis*. The Kew authorities, whose attention has been drawn to these omissions, inform us that they will now be included in the eleventh Supplement.

100,000 gallons in about 1935 (Penfold, 1935. *Aust. J. Pharm.*: p. 29) to 190,192 gallons (valued at £A 208,282) in 1941-42 (Penfold & Morrison, *loc. cit.*). These exports were made up of 65 to 70 per cent. of medicinal oils and 30 to 35 per cent. of industrial oils. The amount of medicinal oils contributed by different species was approximately like this: *E. polybractea* 33 per cent., *E. sideroxylon* 20 per cent., *E. leucoxylon* 20 per cent., *E. australiana* and *E. dives*, var. "C" 20 per cent., and the remaining species 6 per cent.

According to Penfold & Morrison (*loc. cit.*), Smith-White [1940. *J. Aust. Inst. agric. Sci.*, 6(4)] has shown that in Australia there is a return of about 12s. 6d. per acre from land under oil-yielding eucalypts as compared to 1s. per acre from land under grazing by sheep and cattle.

**Summary and recommendations** — It will now be seen that out of about 500 species of *Eucalyptus* 90 have been introduced into India with varying degree of success at different places. Almost all the introduced species are arboreal in their habit. Although eucalypts are well known for the oil which they contain, it is only a few species which are exploited commercially. In Australia the present-day commercial oils are mostly won from *E. australiana*, *E. citriodora*, *E. cneorifolia*, *E. dives* (type), *E. dives* var. "A", *E. dives* var. "C", *E. dumosa*, *E. elaeophora*, *E. leucoxylon*, *E. macarthurii*, *E. numerosa*, *E. numerosa* var. "A", *E. phellandra*, *E. polybractea*, *E. sideroxylon*, and *E. viridis*. In addition, *E. odorata* and *E. smithii* are also important species. Of all these, only *E. citriodora*, *E. dives*, *E. elaeophora*, *E. leucoxylon*, *E. macarthurii*, *E. sideroxylon*, and *E. smithii* have been tried in India for ornamental or fuel purposes. They do not appear to have attracted any attention in India from point of view of the valuable essential oils, and only casual efforts have hitherto been made to cultivate them. If they have not been successfully cultivated at one place no attempts have been made to try them at other places. Nor has there been any serious attempt to augment their cultivation where they thrive.

Eucalyptus oils are put to wide uses, depending upon the constituents which they contain, and are grouped as under:—

1. Cineole oils for medicinal purposes, as disinfectants, germicides, insecticides, etc.: The medicinal value of the oil depends upon its cineole content, and for this reason

mallee oils have become important. In India, the medicinal oil is prepared from the leaves of *E. globulus*, which yield about 1 per cent. of oil containing about 60 per cent. of cineole. Eucalyptus oil containing less than 70 per cent. of cineole is not acceptable to the British Pharmacopoeia. It is evident, therefore, that the Indian oil has to be rectified to bring up the cineole content to a minimum of 70 per cent., and further requires improvements in those other characteristics in which it differs from the Pharmacopoeial specifications. This means additional cost to make the oil acceptable. Fortunately, there are other species, such as *E. polybractea*, *E. cneorifolia*, *E. australiana*, *E. viridis*, *E. odorata* and *E. smithii*, which yield  $1\frac{1}{2}$  to 4 times the amount of the oil obtainable from *E. globulus*, the oil containing 70 to 85 per cent. of cineole. Except *E. australiana* and *E. smithii* which are trees, all these species have a shrubby growth and the collection of leaves from these mallees is easier. Indeed, the bulk of Australian cineole-rich oils is at present distilled from these mallees, and is up to the B.P. standard. The Indian Pharmacopoeial List Committee have, however, recognized a cineole content of 55 per cent. This is probably because the Indian oil normally does not contain a higher percentage of cineole, but it should not be forgotten that in the field of medicine the public are entitled to the best. Attempts should, therefore, be made to cultivate suitable species in the country so as to produce oil of present B.P. quality. It is, therefore, recommended that the mallees *E. polybractea*, *E. cneorifolia*, *E. viridis*, and *E. odorata*, especially the first-mentioned, should be introduced into India. These mallee oils further contain a constituent, aromadendral (cuminaldehyde, phellandral, and *l*-cryptone), which is reported to possess 5 to 6 times the bactericidal value of cineole.

The essential oil from *E. dives* (type) contains 40 to 50 per cent. piperitone, which is easily convertible into menthol and thymol. The cultivation of this species in India is, therefore, recommended.

2. Perfumery oils: These are the oils in which acetic ester of geraniol, usually associated with the free alcohol, is present. *E. macarthurii* is the best source of geranyl acetate which is present in the oil to an extent of 70 per cent. This is found both in the leaf and bark oil.

Citronellal is another constituent which is valuable in perfumery. The oil of *E. citriodora* consists almost wholly of citronellal (up to 85 per cent.).

Citral occurs in the oil of a few species, and *E. staigeriana* yields a lemon-scented oil containing about 16 per cent. of this important constituent.

All these three species should be cultivated in India.

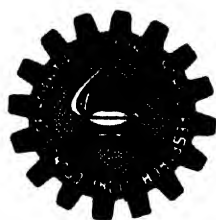
3. Phellandrene oils are used for industrial purposes, such as ore flotation, prevention of scales in steam boilers or motor cars,

and as a general solvent for paints and varnishes, resins, waxes, and certain cellulose lacquers. For these purposes cultivation of species like *E. phellandra* and *E. dives* var. "A" are recommended.

According to Kolachov (*vide* Anon., 1942. *Perfum. essent. Oil Rec.*, **33** : 130), Australia and Russia use phellandrene as an antiknock agent in gasoline. Penfold (1949. *Private communication*), however, informs us that the antiknock properties of phellandrene have not been sustained.

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# Stabilizers for Fats Used in Baking

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FAT is an essential ingredient of most classes of baked goods. It plays an important rôle in the development of flavour and in determining the physical structure of the product. The amount of fat in baked products varies from 3 to 50 per cent and as they are usually packed in containers which do not protect them from air, there is a possibility of the fat turning rancid. The problem of fat stability in baked goods is quite distinct from the stability of fats in their original form. Very little correlation between the keeping qualities of different fats and the stability of baked products made from the same fats has been found to exist. Thus Triebold, Webb and Rudy<sup>1</sup> found that rancid lards produced crackers of better keeping quality than did lards of good quality.

Bohn and Olson<sup>2</sup> observed that the stability of crackers decreased with the increase in the unsaturation of fats. This effect has been attributed to the destruction of anti-oxidants in baking. McKinney and Bailey<sup>3</sup> found that biscuits made with hydrogenated lard are nearly as stable as those prepared from hydrogenated oil although the concentration of natural anti-oxidants in lard is much lower than that in vegetable oils.

Besides factors such as baking temperature, ingredients, metallic contamination during baking, moisture content and condition of storage, the nature and quality of fat are important factors determining the stability of baked goods. Animal fats such as lard and tallow are seldom used in Indian bakeries. Hydrogenated vegetable oils (*Vanaspati*), clarified butter (*ghee*), coconut oil and vegetable oil shortening are generally employed. The natural anti-oxidants present in oils and butter are largely eliminated

during the refining and hydrogenation processes, and even if present, they are inactivated during baking. It is necessary, therefore, to stabilize the fats by anti-oxidants which are stable under the conditions obtaining in the baking process.

The comparative merits of a series of non-toxic anti-oxidants which may find application in the baking industry in India have been studied in these laboratories as a preliminary to the study of fat stability during the baking process. The fats were subjected to "swift stability test"<sup>4</sup> with and without additives and the acid and peroxide values (Wheeler's method<sup>5</sup>) of the resulting products examined. The additives which are found effective as a result of these tests will be selected for further study under actual baking conditions.

## Experimental

The fats were examined and their characteristics are given in Table I.

TABLE I

FAT	ACID VALUE	SAPONIFICA- TION VALUE	IODINE VALUE
<i>Vanaspati I</i>	0.63	188.7	85.9
<i>Vanaspati II</i>	0.35	190.9	62.4
<i>Ghee</i>	5.80	245.3	32.2
Vegetable oil shortening	0.52	189.6	16.4
Coconut oil	6.07	249.7	8.1

The "swift stability test" on the fats was carried out as follows: 20 gm. of the fat were taken in each of 6 test tubes (1"×8") and immersed in a thermostat at 210°F. Air washed with potassium permanganate solution was bubbled through the fat in each of the test tubes at the rate of 2.33 cc. per second. The acid value, peroxide value and smell were determined at noted intervals. The results obtained are given in Table II (Fig. 1).

TABLE II—SUSCEPTIBILITY OF FATS TO OXIDATION

FAT	AERATION TIME, HR.	ACID VALUE	PEROXIDE VALUE	SMELL
1. <i>Vanaspoti I</i>	0	0.63	1.20	Good
"	1.5	3.60	3.40	Good
"	2.5	0.41	4.14	Good
"	3.5	0.26	9.24	Good
"	5.5	0.35	18.50	Good
2. <i>Vanaspoti II</i>	0	0.35	0.73	Good
"	1.5	0.45	5.80	Good
"	2.5	0.42	10.90	Good
"	3.5	0.43	42.80	Slightly rancid
"	5.5	1.56	93.30	Rancid
3. <i>Ghee</i>	0	5.80	0.28	Good
"	1.5	5.24	8.23	Good
"	2.5	5.17	26.8	Slightly rancid
"	3.5	6.21	44.50	Rancid
"	5.5	10.10	80.10	Rancid
4. Vegetable oil shortening	0	0.52	1.30	Good
"	1.5	0.30	1.60	Good
"	2.5	0.64	2.10	Good
"	3.5	0.40	3.01	Good
"	5.5	1.30	6.82	Good
5. Coconut oil	0	6.07	0.07	Good
"	1.5	4.08	0.16	Good
"	2.5	3.93	0.16	Good
"	3.5	2.6	0.42	Good
"	5.5	1.6	0.78	Good

The effect of adding anti-oxidants was next studied. All the anti-oxidants, except N.D.G.A. (nordihydroguaiaretic acid), were incorporated in the fats by direct addition with gentle warming and stirring. N.D.G.A. (0.1 gm.) was dissolved in 10 gm. of refined groundnut oil and an aliquot of the solution incorporated into the fat. The results obtained are given in Tables III-VI. As coconut oil keeps well as such, no attempt was made to study the effect of anti-oxidants on its stability.

TABLE III—VANASPATI I

ANTI-OXIDANT	AERATION TIME, HR.	ACID VALUE	PEROXIDE VALUE
1. ...	nil	0.6	1.2
2. ...	3.5	0.2	9.2
3. Alcoholic extract of <i>anis</i> seed (solid 0.1%)	3.5	0.2	8.9
4. Sesame seed powder (0.1%)	3.5	0.2	7.3
5. Egg lecithin (0.1%)	3.5	0.1	7.5
6. Soya-bean lecithin (0.1%)	3.5	0.3	7.2
7. <i>Kamala</i> dye (0.1%)	3.5	nil	8.1
8. <i>Kamala</i> dye (0.1%) + tartaric acid (0.02%)	3.5	0.3	7.4
9. <i>Kamala</i> dye (0.1%) + citric acid (0.02%)	3.5	0.1	7.7
10. N.D.G.A. (0.01%)	3.5	0.1	9.0
11. N.D.G.A. (0.01%) + citric acid (0.02%)	3.5	0.2	8.1
12. Wheat germ oil (0.1%)	3.5	0.2	8.5
13. Wheat germ oil (0.1%) + citric acid (0.02%)	3.5	0.1	1.4

TABLE IV—VANASPATI II

ANTI-OXIDANT	AERATION TIME, HR.	ACID VALUE	PEROXIDE VALUE
1. ...	nil	0.3	0.7
2. ...	3.5	0.4	42.8
3. Alcoholic extract of <i>anis</i> seed (solid 0.1%)	3.5	0.1	27.0
4. Sesame seed powder (0.1%)	3.5	0.1	24.0
5. Oat powder (0.1%)	3.5	0.2	18.0
6. Egg lecithin (0.1%)	3.5	0.1	19.2
7. Soya-bean lecithin (0.1%)	3.5	0.5	16.0
8. <i>Kamala</i> dye (0.1%)	3.5	0.2	33.0
9. <i>Kamala</i> dye (0.1%) + tartaric acid (0.02%)	3.5	0.2	32.0
10. <i>Kamala</i> dye (0.1%) + citric acid (0.02%)	3.5	0.3	32.8
11. N.D.G.A. (0.01%)	3.5	0.4	28.2
12. N.D.G.A. (0.01%) + citric acid (0.02%)	3.5	0.1	7.6
13. Wheat germ oil (0.1%)	3.5	0.1	31.7
14. Citric acid (0.02%)	3.5	0.3	20.2
15. Wheat germ oil (0.1%) + citric acid (0.02%)	3.5	0.3	4.8

TABLE V—GHEE

ANTI-OXIDANTS	AERATION TIME, HR.	ACID VALUE	PEROXIDE VALUE
1. ...	nil	5.8	0.3
2. ...	3.5	6.2	43.5
3. Alcoholic extract of <i>anis</i> seed (solid 0.1%)	3.5	4.7	8.5
4. Sesame seed powder (0.1%)	3.5	4.9	7.6
5. Egg lecithin (0.1%)	3.5	2.5	26.7
6. Soya-bean lecithin (0.1%)	3.5	5.5	1.9
7. <i>Kamala</i> dye (0.1%)	3.5	4.9	2.7
8. <i>Kamala</i> dye (0.1%) + tartaric acid (0.02%)	3.5	4.5	2.2
9. <i>Kamala</i> dye (0.1%) + citric acid (0.02%)	3.5	5.4	2.5
10. N.D.G.A. (0.01%)	3.5	6.1	37.3
11. N.D.G.A. (0.01%) + citric acid (0.02%)	3.5	3.8	20.0
12. Citric acid (0.02%)	3.5	4.2	25.1
13. Wheat germ oil (0.1%)	3.5	5.2	30.3
14. Wheat germ oil (0.1%) + citric acid (0.02%)	3.5	5.0	0.7

TABLE VI—VEGETABLE OIL SHORTENING

ANTI-OXIDANT	AERATION TIME, HR.	ACID VALUE	PEROXIDE VALUE
1. ...	nil	0.5	1.3
2. ...	3.5	0.4	3.0
3. Egg lecithin (0.1%)	3.5	0.3	2.7
4. Soya-bean lecithin (0.1%)	3.5	0.3	1.5
5. <i>Kamala</i> dye (0.1%)	3.5	0.3	2.1
6. <i>Kamala</i> dye (0.1%) + tartaric acid (0.02%)	3.5	0.4	1.3
7. <i>Kamala</i> dye (0.1%) + citric acid (0.02%)	3.5	1.3	1.4
8. N.D.G.A. (0.01%)	3.5	0.5	2.3
9. N.D.G.A. (0.01%) + citric acid (0.02%)	3.5	1.1	1.3
10. Wheat germ oil (0.1%)	3.5	0.4	1.7
11. Citric acid (0.02%)	3.5	1.2	1.8
12. Wheat germ oil (0.1%) + citric acid (0.02%)	3.5	0.3	1.7

### Discussion

From the data in Table II it will be seen that aeration affects the acid values of different fats to varying extents, while the peroxide values increase continuously during the aeration period. This is in accordance with the finding of Kaloyereas<sup>6</sup>. The peroxide value has, therefore, been taken as a measure of rancidity in this investigation. The fact that the peroxide value after 5½ hours' aeration of *Vanaspati II* increased much more than that of *Vanaspati I*, although the iodine value of the former was less than

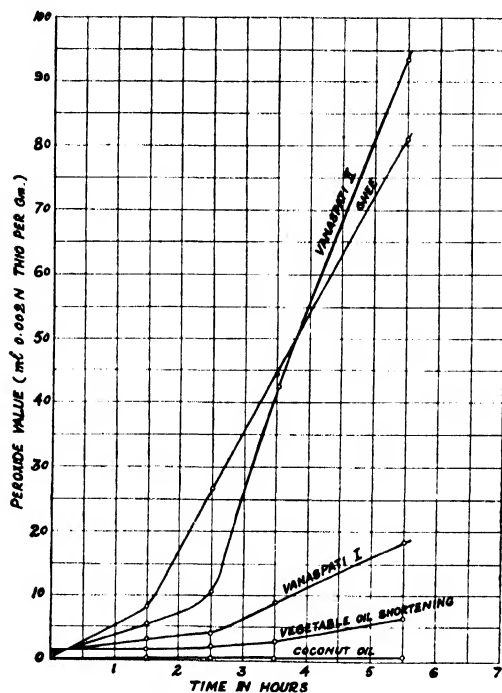


FIG. 1—A COMPARISON OF THE RATE OF OXIDATION OF FIVE FATS SELECTED FOR TESTS.

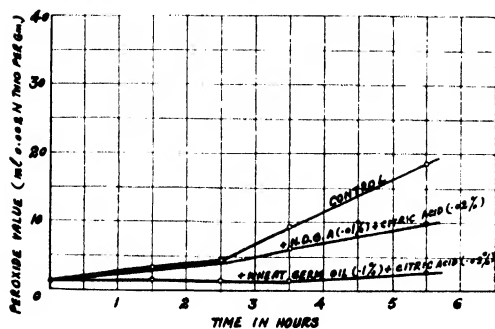


FIG. 2—CHANGE OF PEROXIDE VALUE OF VANASPATI I WITH SOME EFFECTIVE ANTI-OXIDANTS.

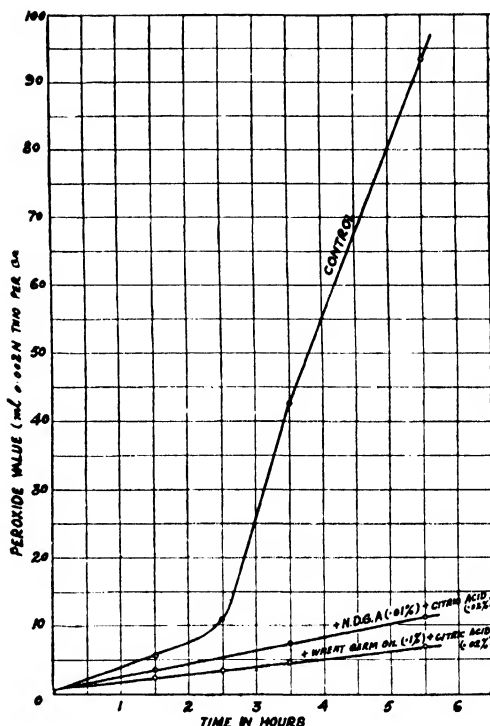


FIG. 3—CHANGE OF PEROXIDE VALUE OF VANASPATI II WITH SOME EFFECTIVE ANTI-OXIDANTS.

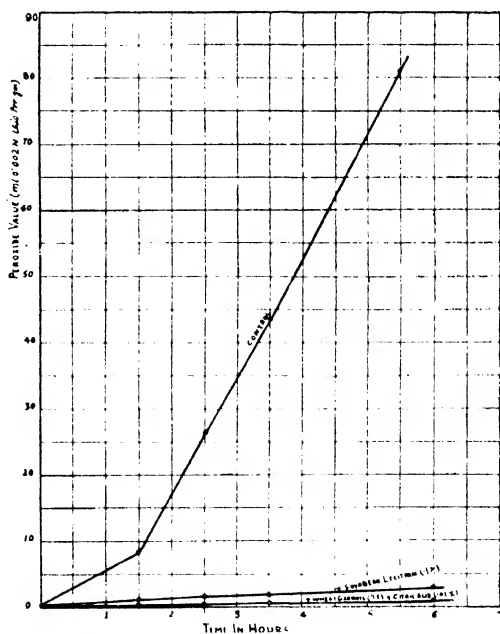


FIG. 4—CHANGE OF PEROXIDE VALUE OF GHEE WITH SOME EFFECTIVE ANTI-OXIDANTS.

that of the latter, goes to show that besides iodine value, other factors such as the fatty acid composition and the pre-treatment of the fat also influence the development of rancidity.

Five anti-oxidants have been selected for further tests as a result of the investigation. They are :

1. Wheat germ oil (0.1%) + citric acid (0.02%).
2. N.D.G.A. (0.01%) + citric acid (0.02%).
3. Soya-bean lecithin (0.01%).
4. *Kamala* dye (0.1%) + tartaric acid (0.02%).
5. *Kamala* dye (0.1%) + citric acid (0.02%).

The order of their effectiveness against *Vanaspati I* and *II*, *ghee* and vegetable oil shortening are recorded in Table VII. The changes in peroxide values of the fats as a result of aeration in presence of the selected anti-oxidants are represented in Figs. 2-5.

TABLE VII

ORDER OF EFFECTIVENESS

<i>Vanaspati I</i>	(1)	(2)	(3)
<i>Vanaspati II</i>	(1)	(2)	(3)
<i>Ghee</i>	(1)	(3)	(5)
Vegetable oil shortening	(2)	(4)	(5)

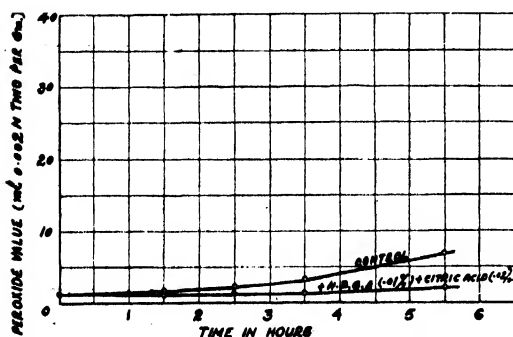


FIG. 5 — CHANGE OF PEROXIDE VALUE OF VEGETABLE OIL SHORTENING WITH N.D.G.A. + CITRIC ACID.

From the above it will be seen that wheat germ oil is the most effective anti-oxidant for *vanaspati* products and *ghee*. N.D.G.A. and soya-bean lecithin follow wheat germ oil in their effectiveness. *Kamala* dye has been found quite effective for *ghee* and vegetable oil shortening. As a matter of fact, vegetable oil shortening can be protected equally well by N.D.G.A., *kamala* dye and wheat germ oil with various synergists. This may be due to the different constitution of the shortening as compared to natural fats. If the colour of the *kamala* dye is not objectionable, it can be employed as an effective anti-oxidant for these fats. All the anti-oxidants except soya-bean lecithin have to be reinforced by synergists such as citric and tartaric acids.

From Fig. 1 it will be clear that there is little increase in the peroxide value of coconut oil after 5½ hours' aeration. The increase is very slight in the case of vegetable oil shortening. This is due to the very low iodine values of these two products. The peroxide value of *ghee* increases rapidly after the initial 1½ hours period. In the case of the two *vanaspati* products an increase is obtained only after 2½ hours. *Vanaspati* products are refined fats, and are sometimes protected by anti-oxidants before they leave the factories.

#### Acknowledgement

The authors record their grateful thanks to Dr. S. Siddiqui for his keen interest in this investigation.

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# A Gene-Determining Growth Rate in Yeast

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THE essential criterion for the selection of a suitable strain for compressed yeast manufacture is the growth rate. Is it possible to plan a breeding programme for continuous improvement? Necessarily, the method of approach for production of improved strains for compressed yeast should be different from that for brewing and distillery industries. No programme of investigations could be planned on the basis of published work on yeasts. Is the growth rate of a strain determined by specific genes or by particular chromosomal or genic constitution? The genetical investigations on yeast by Winge<sup>1</sup> and Lindegren<sup>2</sup> were with strains whose chromosome numbers were unknown. The demonstration of polyploidy in yeast<sup>3,4</sup> has invalidated some of the assumptions of the earlier investigators. The terms "haploid" and "diploid" that one comes across in the literature on yeasts have no relation to the actual chromosome constitution, since the "haploid" of an autotetraploid is really a diploid. Any conclusion based on the assumption that all "haploids" have only one set of genes are necessarily inexact. It appears, therefore, that the earlier investigators had no criteria to distinguish between chromosomal and gene mutations.

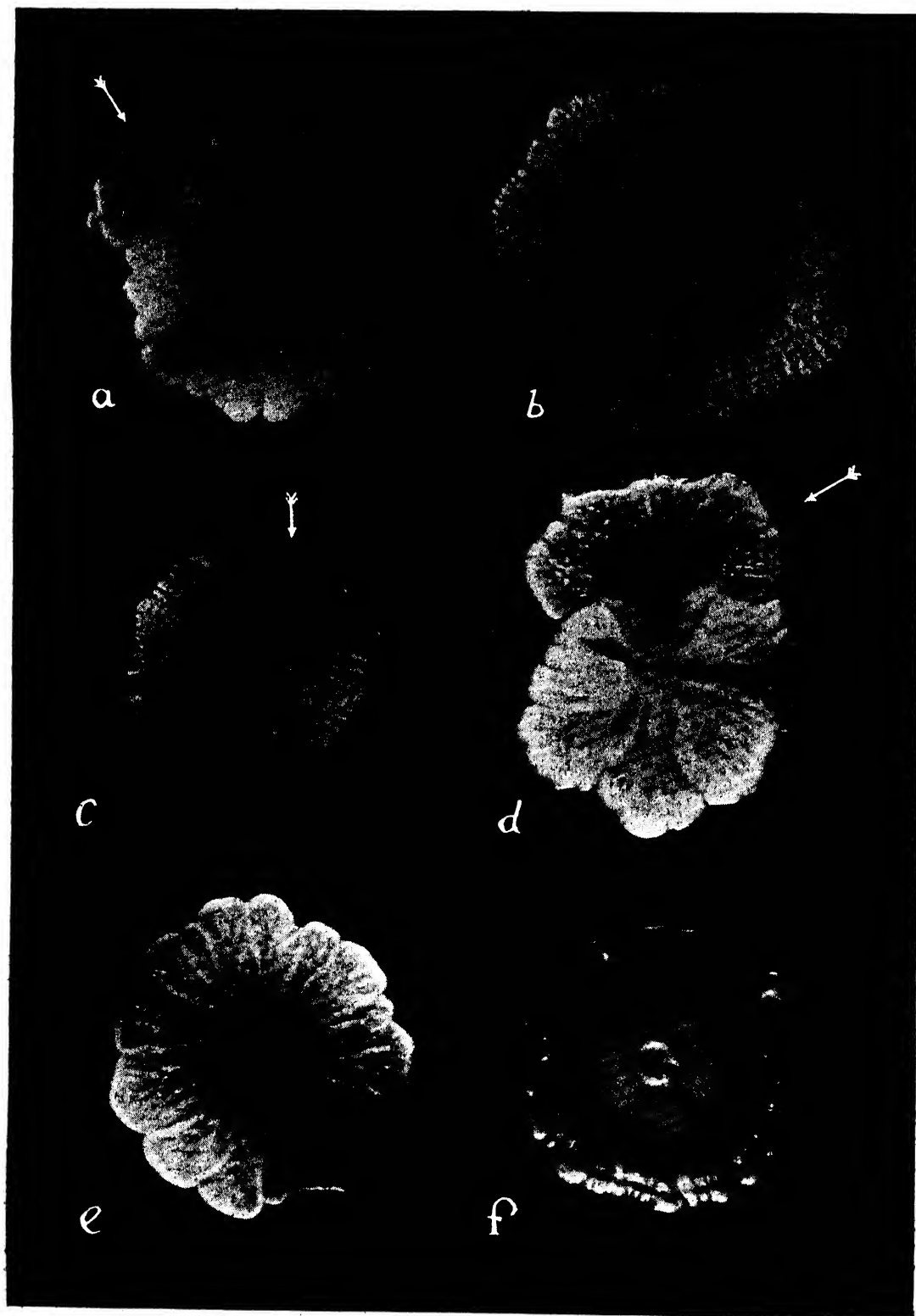
Investigations in this laboratory indicated that the viability and growth rates of the various mutants differed from season to season<sup>5</sup>, that even strains homo- and heterozygous for the same allele show differences under the same environmental conditions<sup>6</sup>, and that doubling of the chromosomes results in a shortening of the generation time and the complete suppression of the second cycle of growth<sup>7</sup>. These investigations never gave an idea that growth rate may be determined by specific genes. The discovery that such growth genes exist was, therefore, of considerable significance.

## Observations

In March 1947 when the control strain, *BY1*, was exposed to a mercury arc for an hour at a distance of 90 cm., the resultant colony showed some peculiar sectors (FIG. 1a). Though these sectors had an identical type of sculpturing as the main colony, their shape indicated that the cells composing them ought to have a very high growth rate. There is a seasonal change in the characteristics of the giant colony of the control and in July 1947 the outer half of the colony had a rough sculpturing (FIG. 1b). Active cells from the above culture, kept at the same distance as before, were exposed to a mercury arc for 3 hours, and a colony was grown from the treated culture. From the appearance of the colony illustrated in FIG. 1c, ultra-violet light seems to have induced two changes. Irradiation had induced tetraploidy and produced a mutation in the cell resulting in the production of colonies having a completely rough surface. On the basis of evidence from spontaneous reverse mutations it was surmised that the *Rough I* type (FIG. 1b) should be heterozygous for the *rough* allele, having the genic constitution, *rough/smooth*. In a previous contribution<sup>8</sup> a reverse mutation to the entirely *smooth* condition by irradiation was demonstrated. FIG. 1c indicates a change to the completely *rough* type. It is reasonable to conclude that the following changes should have occurred.

1. *Rough I*  $\left\{ \begin{array}{l} \text{Rough} \rightarrow \text{Mutation} \rightarrow \text{Smooth} \\ \text{Smooth} \longrightarrow \text{Smooth} \end{array} \right\}$  *Smooth I* (FIG. 2, a & b)
2. *Rough I*  $\left\{ \begin{array}{l} \text{Rough} - \\ \text{Smooth} - \text{Mutation} \rightarrow \text{Rough} \end{array} \right\} + \text{Rough} \left\{ \begin{array}{l} \text{Rough} \\ \text{Smooth} \end{array} \right\}$  *Rough II* (FIG. 1c.)

From the shape of the sector it would be evident that the tetraploid has a higher growth rate than the rest of the cells of the colony. A comparison of the sectors in FIGS. 1a and 1c would indicate that the



sector in the former has a much higher growth rate than the tetraploid itself. The sculpturing in the sector in Fig. 1a was identical with that of the main colony and hence it was concluded that the increased growth rate may be gene determined.

After exposure of the active *Rough 1* type of cells (Fig. 1b) of the control to a mercury arc for 4 hours, the treated culture was grown for 24 hours in wort and then plated. 9 types were selected from the plates and these have been under constant observation for the past 8 months. Among these, only the strain *BYU2* has repeatedly given rise to sectors similar to that illustrated in Fig. 1a. The sculpturing of the sectors may be identical with that of the colony (Fig. 1a) or may be different. In Fig. 1d it would be observed that the sector has a different appearance.

From observations on a continuous series of giant colonies of the above strain, it appears that the mutation producing a rapidly growing sector has no relation to the nature of sculpturing of the colony. In fact, the evidence indicates that independent mutations are occurring at two different loci. It has, however, not been possible to distinguish between the homo- and heterozygous expressions of this gene. The strain *BYU2*, like the control *BY1*, shows a seasonal variation in the characteristics of its giant colonies when grown at room temperature. In September-October 1947, when spontaneous reverse mutations were first observed in the control, the colony of *BYU2* had the appearance illustrated in Fig. 1e. That irradiation had produced a genic change would be evident from the different characteristics of the colonies illustrated in Fig. 1b, 1c and 1e. In November-December a new allele was observed (Fig. 1f) which was succeeded by the *smooth* types in January 1948 (Fig. 2a and 2b). In April-May-June 1948 when the control began to change from the *smooth* to the *rough* type, similar changes

were observed in *BYU2*. While the main colony (Fig. 1d) was smooth, the sector had vein-like sculpturing.

A sample from the sector was inoculated into a tube of wort, and a fresh giant colony grown from the above had the appearance illustrated in Fig. 2c. The general appearance of the colony was similar to that illustrated in Fig. 1a. This particular mutant was succeeded by a more stable one having a pronounced vein-like sculpturing (Fig. 2d). The curious fact was that the control during the same period gave rise to colonies (Fig. 2e) indistinguishable from the above on the same type of medium.

### Discussion

From the observations detailed above it would be apparent that mutations are occurring at two loci independent of one another. While the mutation producing a cell possessing a high growth rate is an isolated phenomenon, the locus determining the nature of sculpturing of the colony seems to be highly mutable. Thus it is possible to get strains having identical colony appearance, but widely differing in their growth rate.

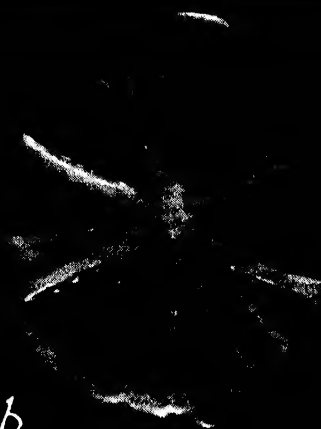
Is the growth gene mutating to a hypermorphic state? Timofeef-Kessovsky<sup>9</sup> seems to have demonstrated such a possibility from X-ray experiments. Mutations in the positive direction are very rare. Muller<sup>10</sup> states: "As for the question, why further 'positive' steps beyond the normal are not observed — from normal to 'hypermorphic' type, i.e. one showing opposite deviation from that of the ordinary mutant — the answer is that such changes doubtless do occur, too, perhaps with a frequency similar to that of the reverse mutations involving origination of the normal itself from a hypermorphic mutant, but they could seldom be observed". Since the terms "hypo-" and "hypermorphs" are used with reference to the standard wild type, any assertion that the extraordinarily high rate of growth of the mutant *BYU2S* is a mutation in the positive direction, would be justifiable only if we know what exactly is the behaviour of the standard wild type growth gene. There appears to be another complicating factor also. The dominance of any gene has been shown to depend on the genotypic background.<sup>11, 12</sup> The genes in domestic fowls, which are dominant over the wild type allelomorphs, become recessive when transferred to a wild genotypic milieu.

← FIG. 1 — (a) CONTROL, *BY1*, GROWN IN BARLEY-MALT-AGAR AFTER EXPOSURE FOR 1 HOUR TO A MERCURY ARC; 30-DAY GROWTH, 3.7 CM. (b) *BY1*, *Rough 1* TYPE OF COLONY; 16-DAY GROWTH, 3.1 CM. (c) CONTROL, GROWN IN BARLEY-MALT-AGAR AFTER EXPOSURE FOR 3 HOURS TO A MERCURY ARC; 9-DAY GROWTH, 2.5 CM., JULY 20, 1947. (d) *BYU2* GROWN IN RAGI-MALT-AGAR; 20-DAY GROWTH, 2.6 CM., MAY 25, 1948. (e) *BYU2* GROWN IN RAGI-MALT-AGAR; 14-DAY GROWTH, 3.7 CM., OCT. 10, 1947. (f) *BYU2* GROWN IN RAGI-MALT-AGAR, 25-DAY GROWTH, 2.5 CM., DEC. 10, 1947.

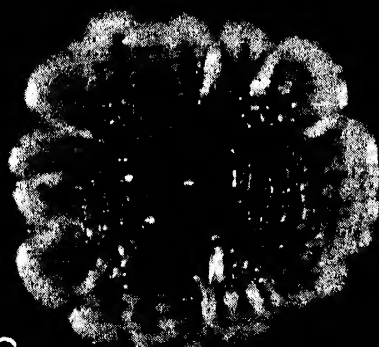




*a*



*b*



*c*



*d*



*e*

Since we do not know the original history of the control strain, *BY1* itself, it would be hazardous to identify any particular type as the standard.

The isolation of a new strain with a shorter generation time naturally raises the question whether further improvement in its performance could be effected. The obvious mode of approach is to induce tetraploidy with acenaphthene. When the tetraploid isolated after treatment of the control with acenaphthene has a shorter generation time<sup>7</sup> the doubling of the chromosomes in the strain, *BYU2S*, should produce a new one having a very high growth rate. The tetraploids studied in this laboratory show improvement over the control in several directions<sup>13, 14</sup>. The most important fact, however, is that doubling the chromosomes and hence the gene dosage is conducive to stability in the vegetative condition since mutations of any one of the genes would not usually find any phenotypic expression at all.

How far these expectations would be realized can be judged only by future experiments.

### Summary

1. Growth genes appear to exist in yeasts.
2. A giant colony grown from a culture exposed to a mercury arc for an hour showed some peculiar sectors. The shape of these sectors indicated that the cells composing them ought to have a high growth rate. A comparison showed that the

new type of sectors had a higher growth rate than even the tetraploid.

3. Spontaneous and periodic appearance of such sectors was observed in a strain, *BYU2*, isolated after 4 hours' ultra-violet irradiation.

4. The sculpturing of the new sectors may be identical with that of the colony or may be different.

5. The seasonal changes in the sculpturing of the giant colonies of the rapidly growing strain are illustrated.

6. It is possible to get strains having identical colony appearance but widely differing in their growth rate.

7. Doubling of the chromosomes should improve not only the stability but also the growth rate.

### Acknowledgement

The authors are grateful to the Council of Scientific & Industrial Research for generous financial assistance.

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← FIG. 2 — (a) *BYU2* GROWN IN RAGI-MALT-AGAR, 15-DAY GROWTH, 2.4 CM., JAN. 7, 1948. (b) *BYU2* GROWN IN RAGI-MALT-AGAR, 15-DAY GROWTH, 2.6 CM., JAN. 5, 1948. (c) *BYU2S* GROWN IN RAGI-MALT-AGAR, 11-DAY GROWTH, 1.8 CM., JUNE 5, 1948. (d) *BYU2S* GROWN IN RAGI-MALT-AGAR, 12-DAY GROWTH, 3.2 CM., JULY 2, 1948. (e) CONTROL, *BY1*, ISOLATED ON DEC. 14, 1945; GROWN IN RAGI-MALT-AGAR, 14 DAY GROWTH, 3.3 CM., APRIL 29, 1948.

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# Experiments in Mineral Khaki—Part III

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THE present study relates to the effect of pre-treatment and after-treatment of the processed material, on the final shade and fastness properties of mineral khaki. In the pre-treatment, the material originally scoured was subjected to the action of wetting-out and swelling agents and also of detergents. The after-treatments consisted of subjecting the material to pressure boil in a small kier and scouring for definite periods with such agents as sodium silicate, which form complex silicates of chromium and iron. The treated fabrics were expected to show superior fastness properties.

In view of the difference in shade and feel and the varying fastness properties of dyed samples it was of interest to subject them to a microscopic study. The study would throw light on the nature of deposition of the pigment on individual fibres, and provide data on the penetration of the pigment particles inside the body of the fibre. Observations were made with a high power microscope and photomicrographs\* were taken. A study of the samples under polarized light was also carried out with a view to obtaining information as regards absorption by fibres of heavy metals from solutions of their salts (Fig. 1).

## Experimental

A study was made of dyed samples developed in a caustic soda bath containing

\*The utility of photomicrographs for assessing the penetration of dyes into the body of textile fibres has been referred to by Preston, *Modern Textile Microscopy*, pp. 88 and 212.

various swelling agents. The swelling agents employed were sodium zincate, Triton B and cuprammonium hydroxide.

1. *Sodium Zincate*<sup>1</sup> — That the swelling action of caustic soda on cotton could be increased by a solution of zinc oxide in alkali was observed by Mercer as long back as 1851, and was later utilized by Davidson<sup>2</sup>.

The scoured drill was impregnated with a solution containing 10 per cent reduced dichromate and 2 per cent ferrous sulphate and the dried fabric was then developed in 25 per cent cold caustic soda, the developing bath containing 2, 5, 10 and 15 per cent sodium zincate on volume basis. The developed fabric was then dried, steamed and washed as usual. The samples obtained were then studied.

From Table I it would be noted that the samples had practically no lustre, had rough metallic feel and were neither uniform nor clear. As regards handle, they were similar to those obtained without the use of swelling agents. Though the 2 per cent sample was greener than the one without the swelling agent in the developing bath, with increase of sodium zincate the tone changed to the brownish side.

From a study of fastness properties it will be noted that the samples had the same fastness to light, washing, rubbing and perspiration as before, but the 10 per cent and 15 per cent sodium zincate samples showed a slight deterioration towards perspiration.

2. *Cuprammonium Hydroxide* — The effect of cuprammonium hydroxide on cellulose was discovered by Mercer and Schweitzer<sup>3</sup> who observed that swelling generally preceded

TABLE I — SODIUM ZINCATE AS SWELLING AGENT

CONCN. OF SODIUM ZINCATE %	SHADE	LUSTRE & BRIGHTNESS	FEEL AND HANDLE	APPEARANCE	TINTOMETER READINGS			FASTNESS			
					Red	Yellow	Blue	Washing	Perspiration	Light	Rubbing
2	Greenish khaki	Very little	Metallic rough, fuller	Not clear and uniform	1.4	3.1	2.4	5	3	8	5
5	Loss of green	"	"	"	1.5	3.2	2.4	5	3	8	5
10	"	"	"	"	1.5	3.2	2.4	5	2	8	5
15	"	"	"	"	1.6	3.2	2.4	5	2	8	5

solution. The relationship between the swelling of cotton and the concentration of cuprammonium hydroxide as measured by its copper number was demonstrated by Brownsatt, Farrow and Neale<sup>4</sup>. A solution containing less than 0.25 per cent copper had no appreciable swelling effect.

Cuprammonium hydroxide was prepared according to Clibbens and Geake<sup>5</sup>. Different amounts of the solution were added to the 25 per cent cold caustic soda developing bath on volume basis. The samples obtained were then studied.

From a perusal of Table II it would be noticed that with increase in copper content, the greenish tone became predominant. All the 3 samples showed distinct lustre and had marked clarity and uniformity of shade. The fabric had a slight metallic rough feel and exhibited a fullness of feel, irrespective of the copper content in the developing bath. The samples had the same

general fastness to washing, light and rubbing as before, but the perspiration fastness of all the 3 samples showed considerable deterioration.

*3. Triton B*<sup>6</sup> — This is one of the recently synthesized quaternary ammonium compounds and is capable of swelling cellulose to such an extent as to give a clear viscous solution. Triton B is trimethyl benzoylammonium hydroxide and is generally available as 30 per cent solution below which it does not dissolve cotton cellulose at room temperature.

The impregnated material was developed in baths containing varying amounts of Triton B (2, 5 and 10 per cent on volume basis of the developing bath).

From a study of Table III it will be noticed that all the 3 samples were deeper in shade than the one without Triton B, and that with increase of Triton B the tone slightly went over to the brownish side.



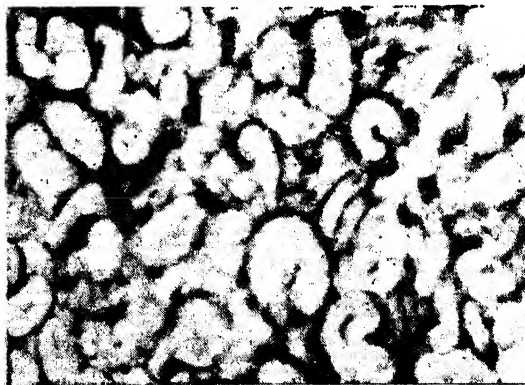
White undyed  $\times 220$ .



Khaki dyed  $\times 220$ .



White undyed  $\times 400$ .



Khaki dyed  $\times 400$ .

The lustre, feel and handle and clarity of the fabric were unaffected by the increase, except that the fabric became full with increasing quantity of Triton B. The dyeings were characterized by lack of uniformity. The general fastness of all the 3 samples to washing, perspiration, light and rubbing was the same, irrespective of the Triton B content (TABLE III). The washing fastness slightly improved, while the rubbing fastness decreased when compared to the sample with no swelling agents in the developing bath. The light and perspiration fastness remained unaffected (the former being excellent, the latter very poor).

**Effect of Pre-treatment on Dyeing**—Throughout the series of experiments described earlier, the grey drill was given only a scouring treatment in the rope form. To study the effect of pre-treatment, the boiled drill was given the following additional pre-treatments prior to dyeing:

The pre-treatments were given on a small jigger of 3-gallon capacity and the fabric was worked in it in open width in the following solutions: (1) containing 2 per cent caustic soda on volume basis at boil for 1 hr. It was then washed free from alkali by hot and cold water, and later with 0.25 per cent acetic acid (30 per cent) to remove

any trace of alkali, washed and then dried; (2) containing 2 per cent caustic soda and 5 per cent sodium zincate on volume at boil for 1 hr. It was then treated as in (1); (3) containing 2 per cent caustic soda and 1 per cent Isapol Brilliant Oil (I.C.I.) (sulphonated ricinoleic acid containing optimum quantities of pine oil) on volume at boil for 1 hr. It was then treated as in (1); (4) containing 2 per cent caustic soda and 1 per cent Aerosol OT which is a powerful wetting agent (Carbide & Carbon Chemicals Corporation) on volume at boil for 1 hr. It was then treated as in (1); (5) containing 2 per cent caustic soda and 1 per cent Triton B on volume for 15 min. at room temperature. It was then treated as in (1); (6) the fabric was treated exactly as above, but treatment was carried out at boil, instead of at the room temperature.

All the differently pre-treated fabrics were then stitched together and processed in the manner described earlier.

From a study of Table IV it would be noted that a pre-treatment in open width considerably influenced the final shade produced. There was a marked increase in the total depth of shade in each case, and on the whole there was a tendency towards a khaki shade. This was in accord-

TABLE II—CUPRAMMONIUM HYDROXIDE AS SWELLING AGENT

CONCN. Cupram. %	Cu %	SHADE	LUSTRE & BRIGHTNESS	FEEL & HANDLE	APPEARANCE	SHADE			FASTNESS			
						Red	Yellow	Blue	Wash- ing	Perspi- ration	Light	Rub- bing
2	0.03	Green khaki	Very lustrous	Fuller and slightly metallic rough	Very clear and uniform	1.3	3.1	2.2	4	2	8	5
5	0.075	Greener	"	"	"	1.3	3.3	2.2	4	2	8	5
15	0.225	"	"	"	"	1.3	3.5	2.4	4	2	8	5

TABLE III—TRITON B AS SWELLING AGENT

CONCN. OF TRITON B %	SHADE	LUSTRE & BRIGHTNESS	FEEL & HANDLE	APPEARANCE	SHADE			FASTNESS			
					Red	Yellow	Blue	Wash- ing	Perspi- ration	Light	Rub- bing
2	Khaki	Considerable lustre	Full, highly metallic	Very clear and not uniform	1.5	3.2	2.6	5	3	8	4.5
5	"	"	Fuller	"	1.7	3.3	2.6	5	3	8	4.5
10	"	"	"	"	1.7	3.3	2.6	5	3	8	4.5

TABLE IV—DIFFERENT PRE-TREATED DYEINGS

No.	SHADE	LUSTRE & BRIGHTNESS	FEEL & HANDLE	APPEARANCE	SHADE			FASTNESS			
					Red	Yellow	Blue	Wash- ing	Perspi- ration	Light	Rub- bing
1	Khaki	Considerable lustre	Soft, fuller	Clear	1.0	3.8	2.4	5	3	8	5
2	"	"	Metallic, fuller	"	1.0	3.8	2.4	2	2.3	8	5
3	Deep khaki	"	Rough, fuller	"	2.0	4.2	2.4	5	3.4	8	5
4	Khaki	"	Metallic, fuller	Clear, uniform	1.8	3.6	2.2	5	3	8	5
5	Light khaki	"	Rough, fuller	"	1.5	3.4	2.1	5	3	8	5
6	Deep khaki	"	Soft, fuller	"	1.8	3.5	2.1	5	3	8	5

ance with the tintometer readings. All the samples showed considerably increased lustre and clarity of shade. The uniformity in general was good but was more pronounced with treatment 4. The pre-treatments 1 and 6 gave a fabric having a comparatively softer feel, while the others gave a metallic rough feel. All the samples were quite full to handle. The dyeings also were of good all-round fastness (TABLE IV).

*Effects of After-treatment*—The mineral khaki samples were submitted to the following after-treatments: (1) the dyed samples were subjected to a simple water-boil in a kier described by Dhingra, Uppal and Venkataraman<sup>7</sup> under a pressure of 30 lb./sq. in. for 3 hr. For this purpose the experimental kier was loaded with 5 lb. bleached yarn and dyed samples were sandwiched in between the yarn, a material-liquor ratio of 1:4 being maintained;

(2) after-treated with 0.8 per cent boiling soap solution for 1 hr. in the open; (3) worked in a 0.5 per cent solution, at boil, of Aerosol OT for 1 hr.; (4) after-treated with a 0.5 per cent solution of Turkey red oil (50 per cent fat content) at boil for 1 hr.; (5) a silicate after-treatment with 1.0 per cent sodium silicate solution at boil for 15 min. was given; (6) after-treatment with 1.0 per cent trisodium phosphate at boil for 1 hr.; (7) processed at boil in a solution containing 0.5 per cent soap and 0.5 per cent soda ash for 1 hr.

The treatments 2 and 7 were carried out in the open. The after-treated samples were washed with cold water, dried, ironed and then examined.

It will be noted from Table V that treatments 2 and 7 resulted in a loss of green. Treatment 3 gave a more greenish shade. The rest showed no change of tone. The

TABLE V — AFTER-TREATED SAMPLES

No.	SHADE	LUSTRE & BRIGHTNESS	APPEARANCE	FEEL & HANDLE	SHADE			FASTNESS			
					Red	Yellow	Blue	Washing	Perspiration	Light	Rubbing
	Original greenish brown	Much lustre	Clear	Fuller, metallic rough	1.4	3.1	2.3	4.5	3	8	5
1	Loss of green, brownish	Increase in lustre	Very clear	Much fuller, metallic rough	1.4	2.9	1.9	5	4.5	8	5
2	"	No change	Clear, no change	Less, fuller soft	1.4	2.9	2.0	5	3.4	8	5
3	"	"	"	More full, metallic rough	1.4	2.9	2.2	5	4	8	5
4	"	Increase in lustre	Very clear	Less full, soft	1.4	2.8	2.0	5	4	8	5
5	Much greener	Decrease in lustre	Loss of clarity	Full, more metallic rough feel	1.3	3.2	2.7	5	4	8	5
6	No change	"	Clear, no change	More full, metallic rough	1.4	3.1	2.2	5	3.4	8	5
7	Loss of green	No change	"	No change	1.4	2.9	2.0	5	3	8	5

TABLE VI

SAMPLE	TREATMENT	OBSERVATIONS	DIA. μ	FASTNESS			
				Washing	Perspiration	Light	Rubbing
1	White, undyed	Many convolutions, no swelling	21.2	...	...	...	...
2	Fabric impregnated with 10% reduced dichromate, 2% FeSO <sub>4</sub> , H <sub>2</sub> O and dried	No swelling; pale green colouration due to salts; brown deposits on surface	22.2	...	...	...	...
3	Above fabric developed in cold 25% caustic soda, dried and washed	Fibres highly swollen with rounding up; pale yellowish shade; clots of surface deposits on some fibres	27.5	4	2.3	8	4.5
4	Impregnated fabric as in (2) developed and steamed	Mature fibres more swollen than in (1); distinct mercerized action; pale yellow uniform shade with surface deposits	24.5	5	3	8	5
5	Same as above, but with 10% sodium zincate in developing bath	More swollen; shade lighter than (4); good uniformity; sheath-like deposits lying beside fibre; large number of surface deposits	26.7	5	2	8	5
6	Same as above, but with 5% cuprammonium in developing bath	Well swollen; large number of surface deposits; good uniformity; mature fibres darker than immature	25.6	5	2	8	5
7	Same as above but with 10% Triton B in developing bath	Good swelling; uniformity not good; mature fibres darker than immature; few surface deposits	24.8	5	3	8	4.5
8	Same as (3) but developing at boil, airing and washing	Shade lighter than others; no surface deposits; less swelling	23.4	4	2	8	5

lustre appeared to increase by treatments 2 and 4, while treatments 5 and 9 caused a decrease in lustre. The rest of the samples showed no change. Similarly, treatments 2 and 4 resulted in clear shades, while treatment 5 resulted in loss of clarity. Treatments 2 and 4 gave a softer handle while the rest of the samples had a more or less metallic rough feel.

It would be noted that the rubbing fastness and light fastness of all the samples were excellent and in every case the washing fastness improved. The perspiration fastness of the original sample, which was 3, was considerably improved by all treatments except the 7, the improvement being more pronounced in the cases of treatments 1, 3, 4 and 5, leading to the conclusion that where improved fastness to washing and perspiration are desired, these treatments could be employed with advantage.

#### Microscopic Examination of Dyed Materials

The microscopic examination was carried out in 3 stages; viz. (a) whole fibre from several sections of yarns of warp and weft were examined for external and internal deposition of salts and penetration of the pigment, resulting in the variation of shades, both under (i) transmitted light, and (ii) polarized light; (b) whole fibres were measured for comparative changes in diameter as a result of the swelling action of the reagents employed in various treatments; (c) sections of yarns were cut to see how deep and how uniformly the pigment had penetrated the yarn and fibre structure.

For the examination of the whole fibres as in (a) and (b), fibres were untwisted from equal numbers of pieces of warp and weft yarns; they were mixed and mounted in tufts of parallelized fibres on glass slides in glycerine. The preparations were covered with a cover-glass and examined under a magnification of 100 dia. for (a) and 500 dia. for (b); a microprojector was used for measuring the diameter. 100 readings were taken with each sample and the average of each noted.

Considerable difficulty was experienced in obtaining and studying the cross-sections of the fibres. Since cotton fibres have a diameter of  $20\mu$  to  $30\mu$  in one direction and only  $2\mu$  to  $5\mu$  in the other direction, stable cross-sections cannot be obtained unless they are less than the measurement of the smaller diameter. Difficulties of cutting cross-sections thinner than  $2\mu$  are well known.

Cross-sections obtained by the cork method were packed together so closely that observations under high power are not practicable with transmitted light. Those obtained by the plate method were found to be unsatisfactory, as they were not sharp and thin. Hence the following technique was employed.

A hand microtome<sup>8</sup> recommended by the *Department of Agriculture, Bureau of Animal Husbandry, U.S.A.*, and which is in use in the Technological Laboratories of the *Indian Central Cotton Committee, Bombay*, was employed. It consists of a bare brass plate with a hole which is packed up with parallel tufts of yarns whose cross-sections are to be cut. It is provided with a swivel arm through which a threaded rod is so journaled that it is able to push the tufts of fibres packed in the hole to a desired distance (about  $15\mu$ ). Before the sections are cut, a drop of cellulose acetate solution ( $5\text{ gm./l}$ ) in acetone is applied on the surface of the yarn pieces showing up through the hole. A safety razor blade is used to cut the section after the thin film of cellulose acetate dried the surface of the yarn pieces. It is necessary to dry the cellulose acetate solution in a desiccator containing phosphorus pentoxide before use.

The cellulose acetate serves as a cementing substance for holding the cut cross-section upright and no attempt was made to dissolve it away. But a mounting fluid with the same refractive index as cellulose acetate was used to keep it out of view while the sections were being examined. Castor oil ( $\Delta=1.48$ ) was used for the purpose.

The photomicrographs taken are shown in Fig. 1.

The following observations were made:

(i) Under transmitted light, the diffusion of pigment inside the body of the fibre was fairly uniform, as shown by the uniformity in the colouration of the fibre and not on the surface only. Also, a distinct swelling of the fibre associated with the removal of convolutions was noticed, resulting in the fibres' assuming the appearance of straight smooth cylinders. These mercerized effects were common in all the khaki-dyed samples, varying more or less according to the treatment given.

It was also observed that the depth of shade and its uniformity varied depending on the sample, so also its fullness. A striking feature of practically all the samples was the peculiar brownish deposits, which

varied in number according to the processing employed. Such deposits were observed even in the fabric impregnated with iron and chromium salts, but not developed, and it may be assumed that these deposits remain even after developing and washing.

Further details of the observations are given in Table VI.

(ii) Untreated cotton fibres showed, under polarized light, a play of brilliant and characteristic colours in mature and half-mature fibres. When the polarizer was rotated through a  $45^\circ$  angle, the immature fibres were extinguished. The play of colours in mature fibres covered practically the whole range of the spectrum, beginning with purple and going to red in fully mature fibres. These colours appeared as short bands across the fibres. In half-mature fibres the play of colours was restricted to blue, yellow and reddish orange, which sometimes appeared as purplish also. Thus, only the middle part of the spectrum was covered. The immature fibres remained distinctly colourless.

The khaki-dyed samples also showed the same colour play in fully mature fibres, but the bands were much broader and the yellow and green colours were noticeably grimy. In half-mature fibres the play of colours was from blue to red to yellow to blue again.

Using the quarter wave-length selenite plate, it was found that the colours in mature fibres of the khaki-dyed samples varied from green to yellow and green to blue with a purplish background, while half-mature fibres showed only yellow to blue bands. The immature fibres looked colourless.

The undyed fibres under similar conditions showed bands of green, yellow and purple colours in a regular pattern while the half-mature fibres showed yellow and blue bands along the walls of the fibres. As before the colours in the khaki-dyed samples lacked brightness.

These observations reveal that fully mature fibres show a maximum deposition of metallic salts, the half-mature fibres less and the immature fibres the least. The presence of these metallic salts leads to the change in shade from the brilliant colours in the undyed samples to rather dirty shades in the dyed samples.

(iii) The measurement of fibre diameter in each sample was attempted using a microprojector and a magnification of 500.

(TABLE VI). The figures indicate the extent of swelling caused by the treatments. It would be noticed from Table VI that the undyed fibres, which had a diameter of  $21.2\mu$ , increased in diameter to  $22.2\mu$  on impregnation with iron and chromium salts. After-development treatments gave rise to considerable swelling. The swelling was least (dia.  $23.4\mu$ ) with hot development and highest (dia.  $27.5\mu$ ) with cold development without steaming. On steaming the swelling decreased (dia.  $24.5\mu$ ).

The introduction of swelling agents in the cold developing bath caused considerable swelling. The zincate sample showed maximum swelling ( $26.7\mu$ ) followed by cuprammonium hydroxide ( $25.6\mu$ ) and Triton B ( $24.8\mu$ ).

(iv) The cross-sections of yarns showed practically uniform colouration, or equal diffusion of the pigment inside the body of the fibres. The rounding up of sections was prominently seen with a distinct swelling, which was due to the mercerizing action of the treatments. These observations are clearly brought out by the photomicrographs of the cross-sections of dyed and undyed samples.

### Summary (Part II & III)

1. *Composition of Impregnating Bath*—The chromium oxide contents of the samples were proportional to the molecular weight of the salt of chromium employed except in the case of sulphate acetate samples. The chromium acetate samples with the highest molecular weight gives the maximum chromium oxide content, followed by sulphate and acetate nitrate samples. The depths are also found to be proportional to the oxide contents. As to the iron oxide content, it was found that for the same amount of ferrous sulphate introduced, the iron oxide deposited was practically the same within experimental limits.

From the alkali-extractable chromium it would be seen that the more complex the chromium compound initially employed for padding, the greater was the alkali-extractable chromium, indicating that it would be preferable to employ simple chromium salts.

The attempt to get chromium as chromium sulphate in the impregnating bath by reduction of dichromate showed that sugar, employed as a reducing agent, has inhibiting action on the oxide deposition and consequently gives lighter shades.



With increase of chromium the perspiration fastness showed a deterioration indicating the sensitivity of the chromium oxide to acids whereas the increase in iron content caused deterioration in the rubbing fastness of the samples.

The acidity of the impregnation bath had much to do with the final shade produced. If the acidity was high (lower than pH 3), the fabric showed tendering after dyeing, and on approaching neutrality (pH higher than 4), the shade gradually showed a change towards the brownish side. The lustre, appearance or uniformity gradually decreased. The perspiration and rubbing fastness deteriorated. It would be desirable to carry out the dyeings at pH 3-4 in order to obtain a dyeing of general all-round fastness.

**2. Optimum Concentration of Alkali in the Developing Bath**—Under the experimental conditions described, the maximum deposition of the metallic constituents was obtained by the use of 25 per cent caustic soda. In view of the wide variations produced in large-scale operations, a steady maintenance of alkali strength between 20 to 25 per cent would result in the least variation of the ultimate shade.

The introduction of cuprammonium and sodium zincate in the developing bath, though giving samples with good fastness to light, washing and rubbing, resulted in dyeings of inferior fastness to perspiration. The Triton B sample remained unaffected. In all the cases the samples developed fuller handle.

**3. Effect of Pre-treatment & After-treatment**—Pre-treatment under suitable conditions is a prerequisite for the production of level shades. Incorporation of wetting out agents of the type of Aerosol OT gives a definite advantage as judged by the increase in depth and levelness of shade produced.

**4. Mechanism & Manner of Deposition**—Chromium and iron salts which were both employed as sulphates exhibited a lack of substantive affinity for the cotton fabric, as shown by the very low quantities of the

oxides fixed by alkali development of the fabric, padded through an impregnation bath, with high initial concentrations.

As the mineral pigment formation is a simple mechanical deposition in the body of a fibre which lacks substantive affinity for these oxides, the levelness and penetration would largely depend on mechanical factors such as the quality of the padding mangle, regulated pressure, uniform dyeing, and so on.

The microscopic study of khaki samples provide interesting data as regards penetration of pigments into the core of the fibre. The degree of maturity of individual fibres affects penetration.

The fastness of mineral khaki to light, washing and rubbing is due to the formation within the fibre of insoluble inorganic complexes of iron and chromium. The perspiration fastness is apparently unsatisfactory. It may be mentioned that the perspiration fastness test is carried out under severe conditions rarely met with in actual practice. Mineral khaki being a metallic pigment is easily affected by the acidity of perspiration. After-treatments impart increased resistance to perspiration.

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# Wear in Producer-Gas Converted Petrol Vehicle Engines

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ONE possible reason for greater wear with producer gas as applied to converted petrol vehicles is that owing to reduced power output, lower gear ratios are more frequently used, which means that the engine makes more revolutions for a given mileage of the vehicle. This increase will be negligible when making long continuous runs on level road for which type of duty these producer-gas converted petrol vehicles are best suited. Another reason is the abrasive action of dust carried along in producer gas. This will be a serious cause of wear in case the filters are not properly designed, serviced and maintained. The exact amount of wear caused in such a vehicle fitted with a well-designed and suitably serviced gas filtration system is not precisely known. The Indian specifications<sup>1</sup>, which are modelled on the Australian specifications<sup>2</sup>, fix the limit of dust concentration in the filtered gas supplied to the engine as 5 mg. per cubic metre. This figure was adopted in the belief that it will probably keep down the abrasive wear within reasonable limits without requiring too elaborate a system of filters. Very little published data is, however, available to confirm or disprove this belief.

Bowden and Kennedy<sup>3</sup>, working in Australia, used Chevrolet, Ford V8 and Bedford trucks, operating on producer gas, using charcoal as fuel and lubricated with S.A.E. 30 oil for experimental road trials. Separate wear figures for petrol and producer gas were determined when using these types of vehicles. The measured wear observed in Ford V8 truck has been found to be comparable to that in Chevrolet and Bedford trucks. Spier and Giffen<sup>4</sup>, in U.K., have carried out extensive bench tests on the performance of a converted petrol engine

with different producer-gas fuels and on the effect of varying compression ratios, etc. The change in viscosity, acid value and percentage ash content of the lubricant were determined by analysing the samples of oil drawn from the sump. The wear of cylinder bore and top piston ring was also determined.

Another reason for increased wear might be the deterioration of lubricating oil or the lubricating system. This aspect can only be investigated by a combined study of oil behaviour and wear. Unlike petrol, such studies with producer gas have been very few. Other causes for wear might possibly be the corrosive effects of certain impurities in producer gas, inadequate protection from atmospheric dust at the air inlet to the gas carburettor, etc. It will require elaborate studies to isolate the effects of each of these factors and suggest remedies. Meanwhile any wear data relating to actual running conditions on Indian roads will go some way towards clearing the existing uncertainty. This is all the more important in view of the general belief that producer gas causes excessive wear. With this end in view, it was felt desirable that certain data collected during routine road tests be utilized to determine the wear caused in the petrol vehicle engine when converted to operate on producer gas under Indian road conditions.

## Experimental

A lease-lend 160" wheelbase 3-ton 1943 model Chevrolet truck fitted with a modified "Simpson" gas plant, as described in our earlier paper<sup>5</sup>, was being used for various departmental trials on producer gas reliability and performance. The vehicle was found to pass when tested in accordance with the

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procedure for road and bench tests as laid down by the *Conference of Producer Gas Technical & Testing Officers, India*<sup>1</sup>. Before starting the test, the engine fitted to the truck was dismantled, thoroughly cleaned and overhauled. Measurements of cylinder bores, big-end bearing diameters and weights of top piston rings were recorded.

The engine was reassembled and the requisite quantity of lubricating oil *S.A.E. 30*, of the correct specifications recommended for use in the engine by the manufacturer, was supplied to the crankcase. The crankcase oil was changed and analysed after approximately every 2,000 miles of run. The samples of oil drawn from the sump were analysed in accordance with the *Standard Methods of Testing Petroleum & Its Products* as adopted by the *Institute of Petroleum Technologists, London*<sup>6</sup>. A simple method was adopted for the analysis of the sludge deposited in the crankcase.

After determining the weight of sludge insoluble in petrol and petroleum ether (60°-80°C.), the residue was ignited to determine the total weight of mineral matter present in the sludge. The weight of mineral matter insoluble in aqua regia and the iron content of the oil as well as the sludge were determined by the usual methods. The results of crankcase oil analyses are summarized in Table I, in which analysis of fresh oil has also been included.

Periodic checks of the filtration system by means of bench tests ensured that filters were working properly and that the dust concentration in the filtered gas was never more than 5 mg. per cubic metre. Rectified spirit and a little petrol were also used as fuels during a part of these trials.

After the truck had completed 9,000 miles of run mostly on producer gas, its engine was dismantled. After recording the condition of various parts, they were thoroughly

TABLE I—CRANKCASE OIL ANALYSES OF CHEVROLET 1943 MODEL EXPERIMENTAL TRUCK ENGINE

	UNUSED S.A.E. 30 LUBRICATING OIL	USED OIL AFTER 4 SUCCESSIVE PERIODS			
		After 1,000 mile run on producer gas	After 2,100 mile run on producer gas	After 2,000 mile run partly on gas and partly on spirit	After 2,000 mile run on producer gas rectified spirit mixture
1. Appearance	Clear and transparent.	Dark brown oil with slight sludge	Dark thick oil with lot of sludge	Dark brown thick with black sludge	Dark thick oil with lot of sludge
<i>Oil Analysis</i>					
2. Sp. gr. at 60°F.	0.885	0.889	0.894	0.897	0.890
3. Acid value	0.03	0.50	0.46	0.50	0.290
4. Viscosity in centi-strokes :					
(i) at 100°F.	125.2	136.4	140.8	162.2	145.0
(ii) at 140°F.	42.9	46.2	47.8	54.8	50.5
(iii) at 210°F.	12.3	12.6	13.2	14.5	13.5
Viscosity index	95.0	89.0	89.0	95.0	95.0
5. Ash content, %	0.008	0.54	0.34	0.96	0.613
6. Fe <sub>2</sub> O <sub>3</sub> , parts per million	6	1,000	51,600	197,000	110,000
<i>Sludge Analysis</i>					
7. Wt. of sludge, gm.		29.876	67.700	22.170	48.980
8. Wt. of mineral matter, gm.		20.465	42.680	15.951	26.790
9. Wt. of material insoluble in aqua regia, gm.		12.865*	35.110*	3.270*	14.207*
10. Wt. of Fe <sub>2</sub> O <sub>3</sub> , gm.		4.166	2.86	9.700	4.620

\* Mostly fine dust sand.

TABLE II—WEAR OF PARTS OF EXPERIMENTAL TRUCK CHEVROLET ENGINE PER 1,000 MILE MEASURED AFTER 9,000 MILES RUN MOSTLY ON PRODUCER-GAS FUEL

CYLINDER No.	1	2	3	4	5	6	AVERAGES	AUSTRALIAN FIGURES*	
								Gas	Petrol
1. Wear of cylinder bore in inches :									
(a) Along shaft	0.00032	0.00030	0.00032	0.00028	0.00030	0.00033	0.00031	0.00029	0.00017
(b) Across shaft	0.00028	0.00018	0.00013	0.00021	0.00021	0.00022	0.00021		
2. Loss in weight of top piston ring, gm.	0.281	0.279	0.297	0.131	0.280	0.321	0.261	0.807	0.164
3. Increase in big-end bearing diameter, in inches	0.00072	0.00063	0.00056	0.00064	0.00069	0.00059	0.00064	...	

\* These values are for Ford V8.

cleaned and weighings and measurements repeated for determining the wear which, after reduction to 1,000 miles run basis, are given in Table II. The corresponding gas and petrol figures of the Australian workers<sup>3</sup> when using Ford V8 truck have been also included for comparison.

### Discussion

Taking into consideration the specific gravity, viscosity, acid value and iron content of the four samples of oil analysed (*vide* TABLE I), in every case the viscosity of the used oil has slightly increased as might be expected in the absence of crankcase dilution, but the viscosity index has decreased only in two out of four cases. The acid value, ash content and the iron content have increased in every case. It is significant, however, to note that iron has increased to a greater extent during the periods in which spirit was partly used as fuel.

There is, however, no evidence of the break-down of the lubricating oil such as to cause failure of the lubricating system. Results of the present investigations generally support the observations of Spiers and Giffen<sup>4</sup>.

As regards the sludge analysis, it is clearly brought out that more than 50 per cent of the sludge in every case consisted of fine sand and dust and very small amounts of iron. The fine sand is likely to have come partly from the producer gas and partly from the atmosphere through the air cleaners. It may be stated that air for the gas-air mixture was supplied through the normal oil bath cleaner serving the petrol carburettor air intake. The crankcase was protected by a separate oil bath cleaner. The total weight of sludge in the sump in each case was apparently unrelated to the mileage covered. As already stated, uniform care was bestowed in the filtration and the maintenance of the gas plant, the variation in the weight of sludge deposit has to be attributed to some extraneous agency unconnected with producer-gas operation. It is most likely that due to reasons discussed in an earlier publication<sup>7</sup>, the oil bath cleaners were not able to control effectively the entry of dust under varying atmospheric dust concentrations. The presence of fine sand dust in the sludge signified wear by abrasion.

As to the measured cylinder wear figures, ordinarily one should expect the measured wear of the cylinder bore to be greater in the direction of the piston thrust than parallel

to the crankshaft. But the results of cylinder bore wear summarized in Table II show that the reverse is the case, a paradox for which no satisfactory explanation yet seems to have been offered<sup>8</sup>. It is most probable that the end play of the crankshaft and of bearings at the two ends of the connecting rod are responsible to some extent in wearing away the sides of the cylinders more than the thrust faces. The average cylinder bore wear observed in the present study is only 0.00026" per 1,000 miles and is of the same order as in petrol engines and is comparable to the figures reported by Australian workers. A high rate of cylinder wear was observed by Spiers and Giffen<sup>4</sup> but the wear was considerably reduced when careful attention was paid to gas filtration and the engine was fitted with chrome iron, centrifugally cast "dry" cylinder liners. The wear in the big ends is reasonable and in any case this could be easily adjusted by varying the shims. The corresponding wear figures have not been reported by Australian workers.

The loss in weight of the top piston ring observed in the present study is more or less uniform in all the cylinders and is not so excessive as observed by the Australian workers with producer gas. Their figures for gas are nearly five times their petrol figures. This difference in the loss in weight of the top piston ring observed by the two teams of workers can be explained when we take into consideration the gas filtration system employed by them. The Australian workers used a cyclone filter followed by two large sisal felt filters in parallel, whereas in the present case the filtration system consisted of two cartridge type of filters containing coir fibres and cotton waste followed by a third cotton cloth bag filter<sup>5</sup>. In an earlier investigation<sup>9</sup>, drill cloth has been shown to be superior to the pressed wool felt when tested for the final stage of producer-gas filtration. Moreover experience gained by the authors while using cyclone filters showed that it was very difficult to remove the last traces of fine dust carried along with producer gas, when using charcoal as fuel, by the use of these filters alone. In order to eliminate this fine dust and to bring the dust content of the gas to within 5 mg. per cubic metre, an additional cotton waste filter of the cartridge type was found to be absolutely necessary. From what has been stated above, it seems most probable that the last

traces of fine dust in the gas after leaving the cyclone filters were not removed by the use of sisal felt filters in the final stage of the gas filtration system. During a discussion in the Producer Gas Standing Research Committee Meeting held in 1947, H. D. Choudhury, Producer Gas Technical Adviser to the Government of Bengal, disclosed that he too had been faced with the difficulty of removing the last traces of fine dust while working with cyclone filters alone.

Deposits on piston crowns and cylinder heads were soft and easily removable. The exhaust valve seats were generally pitted and needed recutting before regrinding. The inlet valve seats were less corroded.

According to Ballard, Nixen and Moore<sup>10</sup>, dust is an important contributor to abnormal wear. Whenever abnormal wear occurred, the analyses of the deposits on pistons and in the ring grooves showed these to contain 5 to 25 per cent of silica. Spiers<sup>11</sup> has found the cylinder head deposits containing nearly 60 per cent of silica when operating the engine on gas. The high rate of cylinder wear observed by him is attributed to the dust content of the gas. According to Spiers<sup>11</sup>, the rate of wear can be reduced materially by improving the gas filtration considerably. During the present investigation nearly more than 50 per cent of the sludge deposited in the crankcase contained fine sand dust. The photomicrographs taken by the Australian workers<sup>3</sup> show that abrasion and fine scouring was greater with gas. This is mostly responsible for the excessive wear of the top piston ring observed by the Australian workers. However, the dust present in the air supplied to the

engine is also partially responsible for the observed wear.

The results of the present tests show that if the gas plant is maintained to satisfy the Indian specifications there is no danger of excessive wear. The presence of large amounts of fine sand in the sludge shows that it will pay to give more attention to air cleaning with a view to keep out atmospheric dust from the engine.

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# Effect of Salts on Soil Permeability & Rectification of Alkali-ridden Soils

L. A. RAMDAS

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SINCE 1939, at the suggestion of the *Indian Council of Agricultural Research*, we have been investigating the physics of sub-soil irrigation and allied problems in our laboratories at Poona. While studying the capillary ascent of water through various soils and its evaporation from the top surfaces of the soil columns, it was noticed that in alkali soils and the *bari* soil of the Punjab there was no trace of wetness or any significant evaporation even months after the commencement of the experiment. On repeating the experiment in glass tubes, it was clear that the water did not ascend through these soils due to the impermeable nature of the soil in the presence of the salt.

On investigating the matter further with the black soil of the Deccan using solutions of various concentrations of lithium and sodium carbonates, oxalic acid and lithium and sodium oxalates and a number of similar substances, the present writer, in collaboration with Mr. A. K. Mallik, observed that the impermeability is associated with the capacity of the colloidal coating of the soil particle to undergo swelling under the influence of these substances. The swelling phenomenon was studied both with individual particles as well as layers of the soil. Again, when the soil swells on wetting and then contracts on drying, it exhibits the well-known phenomenon of crack formation. The variations in these phenomena with the concentration of the salt in solution has been investigated in detail. It is found that the swelling and impermeability go on increasing up to an optimum value of the concentration beyond which chemical solution of the organic fraction of the soil colloids sets in with a consequent restoration of the permeability. These effects are best observed with sodium carbonate where a wide range of concentration is possible owing to the

high solubility of this salt in water. Many of the interesting results obtained by us have been recently discussed by Ramdas and Mallik in a series of papers<sup>1-7</sup>.

It was found that in the case of sodium carbonate solutions the black soil of the Deccan showed a maximum impermeability at a concentration of about 2 per cent, indicating this as the most efficient concentration to be maintained in the moisture within the sides of a canal if seepage is to be prevented.

Apart from the above practical aspect *there is the much larger problem of restoring the permeability of the many millions of acres of alkali-ridden lands of the Punjab, parts of the United Provinces, the Central Provinces and the Deccan which get water-logged during the rainy season or after irrigation.* If these tracts can be rendered permeable to water and their salt content leached out, it will be possible to restore their fertility and thus make plant growth feasible.

In our recent investigations the above aspect has received a special emphasis. Recently Ramdas and Mallik<sup>8</sup> reported that a layer of Poona soil previously treated with sodium carbonate and rendered quite impermeable becomes quite permeable to a solution of sodium chloride.

At the suggestion of the present writer, Mr. P. K. Katti working in our laboratory took up a further detailed examination of this subject. In a recent paper<sup>9</sup> he has shown that the beneficial effect of sodium chloride is lost when water is passed through the soil again. It is noticed that though the soil particles shrink in the presence of sodium chloride, opening out the pore space, they still retain the sodium carbonate with which they have been treated previously. On leaching out the sodium chloride with water the sodium carbonate asserts itself again rendering the soil impervious once

again. Thus the restoration of permeability by sodium chloride is only temporary.

In his latest experiments Mr. Katti observes that if instead of sodium chloride a solution of calcium chloride is placed above a soil layer previously rendered impermeable to water by treatment with sodium carbonate, the soil becomes permeable to the calcium chloride solution which passes readily through the soil layer. What is most interesting is that *if thereafter a layer of water is placed above the soil, the water also percolates through readily*. The "alkali" soil once treated with calcium chloride *remains permeable to water in any quantity*. Our experiments show also that the soil so leached out can support vegetation like a normal soil free from sodium carbonate.

The above results have been verified in the case of the *bari* soil of the Punjab and further experiments with "alkali" soils from other parts of the country are also being undertaken.

The above results are also obtained if barium chloride or strontium chloride is used instead of calcium chloride. The

experiments of Mr. Katti are at present in the laboratory stage, *but it will be obvious that the above results are of great practical importance in reclaiming the "alkali" tracts of our country*.

A fuller account of these latest investigations is being given by Mr. Katti in a detailed paper appearing in the *Proceedings of the Indian Academy of Sciences*.

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## Letters to the Editor

### PAPAVERINE FROM INDIAN OPIUM

THE ONLY RECORD TRACEABLE ON THE subject of occurrence of papaverine in Indian opium is the work of Van Italic and Kerbosch<sup>1</sup> which mentions that "Bengal" opium is totally lacking in this alkaloid, which is present in amounts varying between 0.5 to 1.0 per cent in opium from other sources<sup>2,3</sup>.

In response to an enquiry for papaverine from Indian druggists, the present work was undertaken in order to ascertain whether the finding of the earlier workers holds true for opium grown in every part of India. It was found that small quantities of papaverine can be isolated from Indian opium, at least out of the supplies under the control of the Banaras Agency of the Government of India.

### Experimental

The mother liquor left after the separation of the "Gregory salt" obtained during the recovery of morphine and codeine from opium by the Gregory process was used for the purpose. The following scheme was eventually adopted.

The mother liquor left after the separation of Gregory salts during the manufacture of alkaloids from opium is made alkaline with solid caustic soda to about 2 per cent concentration. The alkaline liquid is then extracted 10 times with  $\frac{1}{20}$ th its volume of benzene each time.

From the benzene extract, strained through a cloth filter, the solvent is distilled off. The dark-brown mass left is treated with 1:1 hydrochloric acid till distinctly acidic, diluted with an equal volume of water,

warmed, treated with about 30 gm. activated charcoal for each gallon of liquid and then filtered.

The cooled filtrate is treated with ammonia until precipitation is complete. The precipitated alkaloids are filtered off, dissolved in 1:1 hydrochloric acid, treated with charcoal as before and filtered. The acid solution of the alkaloids is, if necessary, repeatedly subjected to the previous cycle of operations until no further improvement in the colour can be effected.

The clarified liquor is treated with 10 per cent caustic soda solution until strongly alkaline and the precipitated alkaloids allowed to settle. The liquid is filtered off and the precipitate washed free from alkali. It is then treated with 1:1 acetic acid, warmed and filtered.

The acid solution is treated with ammonia until just alkaline (pH 7.5 to 8) and then buffered with solid sodium acetate<sup>4</sup>. The mixture is allowed to stand for 24 hours in the cold and the precipitated alkaloids filtered off and washed.

The precipitated alkaloids, consisting of narcotine and papaverine (mixed with a little cryptopine), are converted into the acid oxalate by treatment with  $\frac{1}{3}$  per cent oxalic acid and adding alcohol to the solution. The papaverine oxalate which is insoluble in alcohol separates. It is filtered off and converted into the base by treatment with 10 per cent caustic soda solution.

The precipitated base is filtered and repeatedly crystallized from alcohol (90 per cent) until the m.p. remains unaltered at 146°-147°C.

Papaverine recovered by following the method described above answered to the following tests:

1. The base showed a m.p. of 146.5°-147.0°C.; authentic sample of the base obtained from U.K. had m.p. 146°-146.5°C.; mixed m.p. 146°-147°C.

2. A 2 per cent solution of the substance in chloroform showed no optical rotation.

3. 0.2 gm. of the substance converted to the hydrochloride gave a m.p. 220°C. The derivative from the authentic sample had a m.p. of 219°C.; mixed m.p. 219°-220°C.

4. 0.2 gm. of the substance converted into picrate gave a m.p. 186°C.; the m.p. of picrate from the authentic sample was 185°C.; mixed m.p. 185°-186°C.

5. 10 mg. of the hydrochloride (3) dissolved in distilled water (100 cc.) and 3 drops of dil. hydrochloric acid and 5 drops

of a solution of pot. ferricyanide added. There was immediate precipitation of lemon-yellow papaverine ferricyanide.

6. A solution of the hydrochloride gave with sodium acetate a crystalline precipitate of papaverine bases, m.p. 146°C.

7. 0.1 gm. of the substance was converted into acid oxalate with  $\frac{1}{3}$  per cent oxalic acid. The oxalate was sparingly soluble in water; almost insoluble in alcohol; m.p. 200°C. corresponding to that of papaverine acid oxalate.

8. The substance dissolves in conc. sulphuric acid (cold) giving a light pink colour. This light pink colour may be due to the presence of small amounts of cryptopine (which was not separated). The light pink solution turned deep red at 110°C. and violet at a higher temperature. On cooling and adding water, the colour disappeared.

Further work is in progress in order to determine the content and yield of papaverine from opium received from different sources, as also of the comparative value of different methods described in literature for the recovery of the alkaloid.

Our thanks are due to the Government of India, Ministry of Finance, Revenue Division, for permission to publish this note.

*Opium Factory  
Ghazipur  
October 4, 1948*

B. K. MUKHOPADHYAY  
C. PARTHASARATHY

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#### ON THE USE OF LIME SLUDGE AS A STABILIZER IN EARTH CONSTRUCTION

IN CONSIDERING THE POSSIBILITIES OF USING earth as a building material in India it may be pointed out that it would not be economical and practicable to use costly ingredients such as cement and bitumen for soil stabilization. Of the two methods of earth construction, viz. rammed earth walls and adobe block walls, the former is definitely cheaper and quicker (being completed within a week from start), but involves skilled labour and some stabilizing agent such as cement,



since simple rammed walls, although quite strong, are susceptible to weathering by rain. The other method is simpler and better suited for Indian conditions where labour is comparatively cheap and a dry period of 3 to 4 months is available in which the blocks can be dried. Blocks of  $9" \times 6" \times 4\frac{1}{2}"$  size are quite convenient to make in ordinary moulds. However, this type of construction also needs some sort of waterproof plaster. In this connection, work has been initiated at the *Building Research Unit*, Roorkee, and a number of indigenous substances such as gums, resins, straw, sodium silicate, lime, molasses and lime sludge have been tried. Cement was also included for the sake of comparison. It has been found that lime sludge can be successfully used for this purpose.

Lime sludge is a by-product of the sugar industry where quicklime is used for neutralizing the acidity. It is estimated that nearly 4,000-5,000 tons of this substance are thrown out as waste every year from a single factory. Preliminary experiments done on local clayey soil containing about 20 per cent sand, 43 per cent silt and 37 per cent clay indicated that 3-4 per cent of sludge is sufficient to stabilize the soil. It was also seen that whereas the ordinary rammed soil discs disintegrate when kept in contact with

water, those stabilized by 3 per cent sludge stand immersion for weeks. It was found that like cement-soil-mixtures, which are cured in humid atmosphere, the sludge-stabilized blocks also gain in strength under humid conditions. In order to see if the sludge can be used for stabilizing highly calcareous soils such as black cotton soils, similar trials were conducted on 2 samples, one from Poona (42 per cent sand, 31 per cent silt and 28 per cent clay) and the other from Sholapur (40 per cent sand, 31 per cent silt and 29 per cent clay), and the results obtained confirmed these findings.

It had been a practice in olden days in this country to mix *gur* solution with lime for waterproofing plaster. Based on this traditional practice, experiments were carried out on the use of lime-molasses mixtures as stabilizers. It was found that the mixture imparts water-repellent characteristics to the soil, but it was not as good as lime sludge. Mud plaster containing 4-5 per cent lime sludge have been tried on test pillars with promising results. Further work in this direction is in progress.

N. K. PATWARDHAN

*Building Research Unit (C.S.I.R.)*  
*Roorkee*  
*September 20, 1948*

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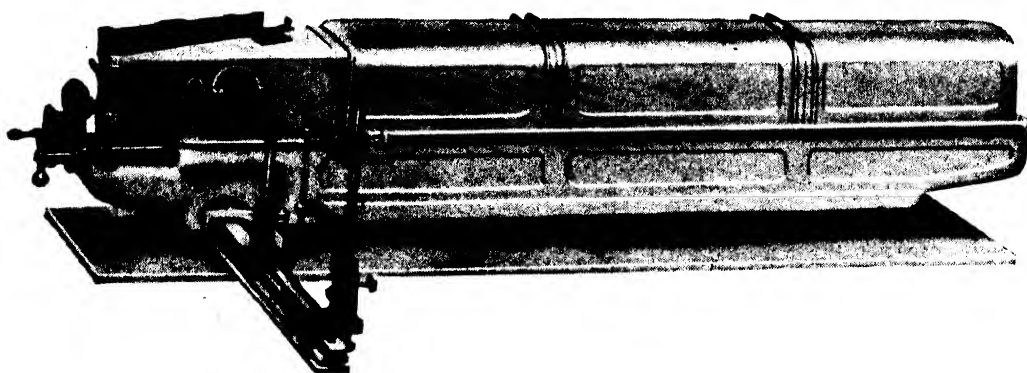
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# The Raman Spectra at Low Temperatures— Diphenyl Sulphide, Diphenyl Methane & Sulphur Monochloride

N. C. MAJUMDAR

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IT was observed previously by Sirkar and Bishui<sup>1</sup> that in the case of some substituted benzene compounds new lines in the low frequency region appear in the Raman spectra when the substances are solidified at low temperatures. It was suggested by them that this phenomenon might be due to the peculiar nature of the molecule so that in the solid state there might be polymeric groups of the substituted benzene molecules giving rise to new lines in the low frequency region. It was thought worth while to investigate the Raman spectra of some compounds in the molecules of which two benzene rings are attached to an atom, because a comparison of the values of the frequency shifts of the lines observed in the case of such molecules with those observed in the case of molecules containing a single benzene ring might throw some light on the origin of these lines. The Raman spectra of diphenyl sulphide and diphenyl methane have been studied with this object in view.

Since the molecule of diphenyl sulphide contains a sulphur atom, the study of the Raman spectrum of another type of molecule containing sulphur atoms but no benzene ring would be interesting in order to understand the significance of the changes which are observed with the solidification of these compounds. For this reason sulphur monochloride ( $S_2Cl_2$ ) has been chosen and its Raman spectra have been investigated in the liquid and solid states.

## Experimental

The arrangement used for the study of the Raman spectra of the substances in the solid state at low temperatures is the same as that used by Bishui<sup>2</sup>. The liquid was put in a sealed pyrex tube which was held vertically inside a transparent Dewar vessel of pyrex glass, the mouth of which was closed with a cork and sealing wax. Through a tube fitting in a hole in the cork, liquid air was introduced by reducing the pressure inside the transparent Dewar vessel with a *Cenco Hyvac* pump connected to a glass tube passing into the Dewar vessel through another hole in the cork. The temperature of the substance in the pyrex tube could be regulated by adjusting the position of the surface of liquid air in the transparent Dewar vessel. A Fuess glass spectrograph having a dispersion of about 13.5 A.U. per mm. in the region of 4046 Å was used. The 4046 Å line of mercury in the spectrogram taken with this spectrograph showed a coma on the short wave-length side, but on the longer wave-length side there was no such coma, and therefore it was possible to detect the presence of new Raman lines lying close to the Rayleigh line.

Diphenyl sulphide was obtained from old stock in this laboratory from a Kahlbaum's sealed bottle. Diphenyl methane was obtained from a sealed bottle from *Eastman Kodak*, Rochester, New York, and sulphur monochloride ( $S_2Cl_2$ ) was obtained from a sealed bottle from *E. Merck*. This

sulphur monochloride was originally found to be deep yellow in colour but on repeated distillation the colour became paler. The 4358 Å line of mercury, however, was extremely weak in the spectrum of the light scattered by the liquid. The absorption of the 5461 Å line was also appreciable because a long exposure was required to photograph the Raman spectrum of the liquid. In the case of sulphur monochloride in the solid state only four hours' exposure was enough to record the Raman lines excited by the line 5461 Å of Hg with fairly large densities. The dispersion of the spectrograph in this region is about 50 Å.U. per mm. Both diphenyl sulphide and diphenyl methane were distilled repeatedly in vacuum and the spectrogram showed no continuous background. Slight weakening of the line 4046 Å due to absorption was observed in the spectrogram due to the light scattered by these liquids.

### Results & Discussion

The spectrograms due to the three substances in the liquid and solid states are reproduced in Figs. 1, 2 and 3. The frequency shifts in wave numbers of the Raman lines observed are given in Tables I, II and III. In the first column of these Tables the data published by some of the previous workers have been included for comparison.

#### Diphenyl Sulphide

It can be seen from Table I that some of the feeble lines observed by Donzelot and Chaix<sup>3</sup> in the case of liquid diphenyl sulphide have not been observed in the present investigation. Of these the line 1155 cm.<sup>-1</sup>

is actually found to be a line at 1165 cm.<sup>-1</sup> and the line 1180 cm.<sup>-1</sup> reported by them seems to be the line 1003 cm.<sup>-1</sup> excited by 4077 Å. Similarly, the line 1475 cm.<sup>-1</sup> excited by 4358 Å is almost superposed on the line 3057 cm.<sup>-1</sup> reported by them. The existence of this line excited by 4046 Å could not be confirmed. It can also be seen that in the case of diphenyl sulphide the frequencies of the lines 1003 cm.<sup>-1</sup> and 1026 cm.<sup>-1</sup> diminish to 995 cm.<sup>-1</sup> and 1015 cm.<sup>-1</sup> respectively with the solidification of the substance and lowering of the temperature to -150°C. These two lines are due to the C-C valence oscillations of the benzene ring in the molecule and the fact mentioned above shows that this bond is weakened a little in the solid state. Further, the line 1584 cm.<sup>-1</sup> shifts to 1578 cm.<sup>-1</sup> and the line 1095 cm.<sup>-1</sup> also shifts to 1085 cm.<sup>-1</sup> and its intensity diminishes considerably at the low

TABLE I — DIPHENYL SULPHIDE (C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>S

LIQUID		SOLID
Δ ν in cm. <sup>-1</sup> Donzelot, P. & Chaix, M. <sup>3</sup>	Δ ν in cm. <sup>-1</sup> At about 32°C. Present author	Δ ν in cm. <sup>-1</sup> At about -150°C. Present author
187 (f)	.....	46 (ls) k
216 (f)	225 (1) e	.....
270 (ff)	265 (1) e	268 (o b) e
410 (fff)	403 (o b) e	400 (o) e
516 (fff)	523 (o) e	.....
614 (m)	618 (2) e, k	618 (o) e
692 (m, large)	695 (2) e, k	697 (o) e
909 (F)	1003 (10) e, k	995 (5) e, k
1023 (m)	1026 (2) e, k	1015 (o) e
1081 (ff)	.....	.....
1092 (aF, b)	1095 (5b) e, k	1085 (ob) e, k
1155 (f)	1165 (1) e	.....
1180 (ff)	.....	.....
1327 (fff)	.....	.....
1440 (fff)	1440 (o)	.....
1475 (fff)	.....	.....
1580 (F)	1584 (6) e, k	1578 (2) e, k
3057 (aF)	3062 (8) e, i, k	3062 (3) e, k

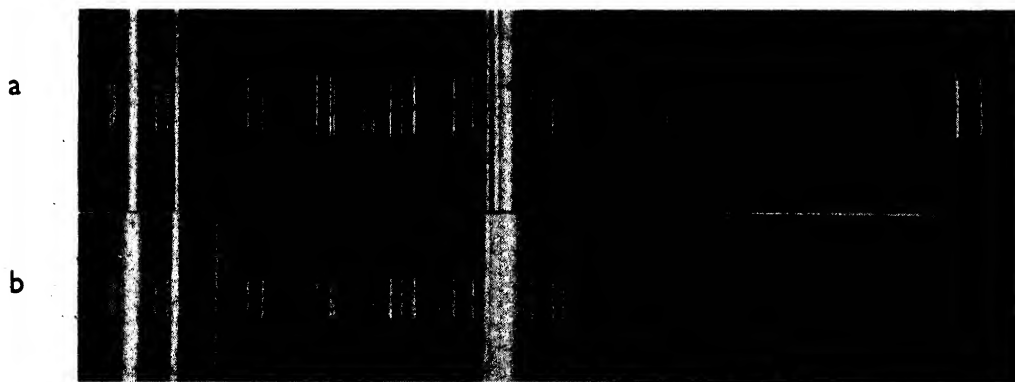


FIG. 1 — (a) DIPHENYL SULPHIDE (LIQUID AT 32°C.). (b) DIPHENYL SULPHIDE (SOLID AT -150°C.).

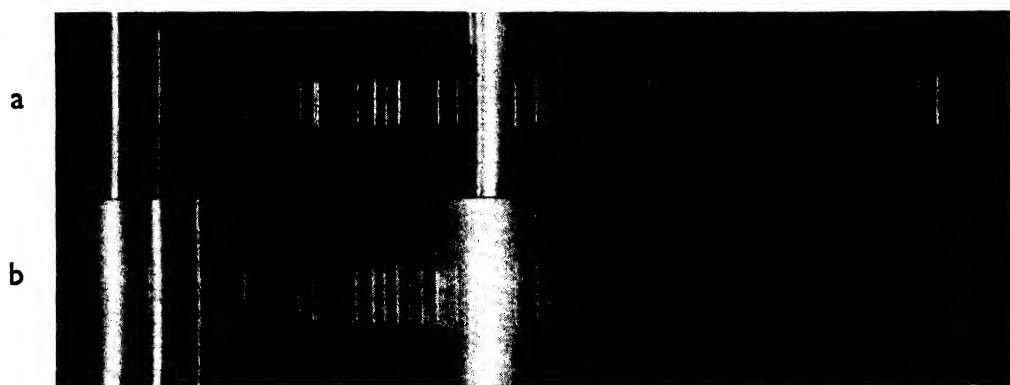


FIG. 2—(a) DIPHENYL METHANE (LIQUID AT 33°C.). (b) DIPHENYL METHANE (SOLID AT -70°C.)

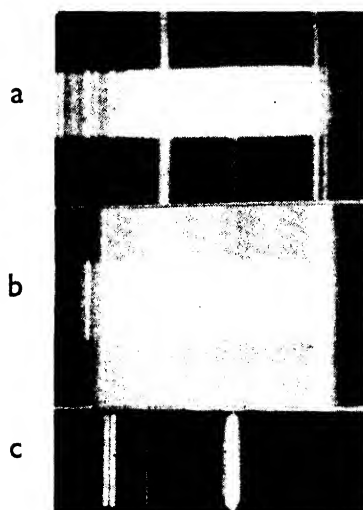


FIG. 3—(a) SULPHUR MONOCHLORIDE (LIQUID AT 33°C.). (b) SULPHUR MONOCHLORIDE (SOLID AT -170°C.). (c) MERCURY ARC (DIFFERENT MAGNIFICATION).

temperature. It is remarkable, however, that the line due to C-H valence vibration of the benzene ring remains in its original position even at  $-150^{\circ}\text{C}.$  and this fact shows that the majority of the C-H groups remain unaffected in the solid state at low temperature.

A new line at  $46\text{ cm}^{-1}$  is observed in the case of solid diphenyl sulphide at the low temperature and it is very sharp. As will be seen later, two such new lines are also observed in the case of diphenyl methane at the low temperature. Recently it has been pointed out by Rousset<sup>6</sup> that in the case of naphthalene and some derivatives of benzene in the solid state the new lines

TABLE II—DIPHENYL, METHANE ( $\text{C}_6\text{H}_5$ )<sub>2</sub>CH<sub>2</sub>

Liquid		Solid
$\Delta \nu$ in $\text{cm}^{-1}$ Donzelot, P. & Chaix, M. <sup>2</sup>	$\Delta \nu$ in $\text{cm}^{-1}$ At about 33°C. Present author	$\Delta \nu$ in $\text{cm}^{-1}$ At about -70°C. Present author
.....	.....	50 (1) k
.....	.....	65 (o) k
199 (f)	196 (2) e	.....
234 (f)	238 (2) e	238 (1) e
286 (fff)	289 (1) e	270 (o) e
457 (fff)	.....	.....
547 (ff)	553 (1) e	.....
599 (fff)	.....	.....
617 (f)	623 (3) e, k	623 (1)
734 (f)	737 (3) e, k	737 (2)
813 (f, large)	817 (3b) e, k	.....
840 (ff)	.....	.....
1001 (f)	1004 (8) e, k	1004 (5) e, k
1026 (m)	1028 (2) e, k	1024 (1) e, k
1155 (f, large)	1154 (o) e	.....
1184 (f, large)	1188 (2b) e	1192 (2) e
1436 (fff)	1425 (o) e	.....
1583 (f)	.....	.....
1601 (m)	1598 (5) e, k	1598 (2) e
2909 (m)	2920 (3) e, k	2920 (o) e
3054 (f)	3060 (10) e, i, k	3060 (3) e, k

TABLE III—SULPHUR MONOCHLORIDE,  $\text{S}_2\text{Cl}_2$ 

Liquid		Solid	
$\Delta \nu$ in $\text{cm}^{-1}$ Venkatesw- aran, S. <sup>4</sup>	$\Delta \nu$ in $\text{cm}^{-1}$ Gerding, H. & Westrik, R. <sup>5</sup>	$\Delta \nu$ in $\text{cm}^{-1}$ At about 33°C. Present author	$\Delta \nu$ in $\text{cm}^{-1}$ At about -170°C. Present author
107 (4d) e	106 D	115 (3) $\pm$ e	115 (3) $\pm$ e
210 (2) e	206 P	202 (3) $\pm$ e	141 (0) e
248 (o) e	245 D	238 (1) e	202 (3) $\pm$ e
449 (6d) e	443 P	454 (8) e	238 (1) e
545 (o) e	537 D	545 (1b) e	349 (3b) e
			442 (8) e
			536 (1b) e
			620 (3b) e
			692 (1) e

in the low frequency region can be explained on the hypothesis put forward earlier by Kastler and Rousset<sup>7</sup> and also independently by Nedungadi<sup>8</sup> that these lines are due to rotational oscillations of the molecule about its three axes in the field of the lattice. It is difficult to say whether this hypothesis

can explain the appearance of the new lines in the low frequency region in the case of the two compounds containing the benzene rings mentioned above, because there is only one new line in one case and two such lines, in the other case. It has to be pointed out, however, that whatever be the nature of oscillation that gives rise to these new lines, some virtual bond is probably responsible for the frequency observed, because some of the lines due to the intramolecular oscillations of the molecule are shifted from their original position when the two substances mentioned above are solidified.

### Diphenyl Methane

In the case of diphenyl methane two new lines at 50  $\text{cm}^{-1}$  and 65  $\text{cm}^{-1}$  are observed in the Raman spectrum due to the solid state and these lines are absent in the Raman spectrum of the liquid. Further, the lines at 289, 1028 and 1188  $\text{cm}^{-1}$  shift respectively to 270, 1024 and 1192  $\text{cm}^{-1}$  with the solidification of the substance. There are slight changes in the positions of a few other lines also. It is interesting to compare the Raman spectrum of diphenyl methane with that of benzene at room temperature. The line 990  $\text{cm}^{-1}$  due to breathing vibration of the benzene molecule is split up into two lines at 1004  $\text{cm}^{-1}$  and 1028  $\text{cm}^{-1}$  and this phenomenon is observed in the case of almost all monosubstituted benzene compounds. The lines 403  $\text{cm}^{-1}$  and 695  $\text{cm}^{-1}$ , however, though present in the Raman spectra of benzene and diphenyl sulphide, are not present in the Raman spectrum of diphenyl methane. It is obvious from this fact that the attachment of the carbon atom of benzene ring to that of  $\text{CH}_4$  alters the frequencies of these vibrations while such an attachment to the sulphur atom in diphenyl sulphide does not affect these frequencies. The behaviour of some of the prominent lines of diphenyl methane on solidification of the liquid is, however, quite different from that of the same lines in the case of diphenyl sulphide. For instance, in the former case the lines 1004 and 1598  $\text{cm}^{-1}$  remain in their original positions in the Raman spectrum due to the solid while these lines shift slightly to lower frequencies in the latter case. It is thus evident that the C-S bond in the latter case is affected primarily with the solidification of the substance and this change in the strength of the C-S bond affects indirectly the frequencies of the vibrations of the

benzene ring mentioned above. It is not unlikely that the appearance of the new lines in the solid state in both these cases may be due to the same cause as is responsible for the change in the frequencies of some of the Raman lines mentioned above.

### Sulphur Monochloride

Sulphur chloride attracted the attention of a large number of workers previously. Venkateswaran<sup>4</sup> showed that the number and frequencies of the Raman lines could be explained by assuming the form by Cl-S-S-Cl of the molecule and that the view held by previous workers was not correct. Mizushima and Morino<sup>9</sup> pointed out that the finite value of the permanent electric moment of the molecule cannot be explained by assuming that the molecule Cl-S-S-Cl has a trans-configuration. They, therefore, suggested that the plane through one of the S-Cl group might make an angle of 90° with the plane through the other S-Cl group in this molecule. Gerding and Westrik<sup>5</sup> have shown that of the five Raman lines of  $\text{S}_2\text{Cl}_2$  in the liquid state, three are totally depolarized and two are polarized, and they have concluded from these results that there is hindered rotation about the *cis* position of the Cl-S-S-Cl structure of the molecule. In the present investigation also only five lines have been observed in the Raman spectrum of  $\text{S}_2\text{Cl}_2$  in the liquid state, and the solid state at about  $-170^\circ\text{C}$ . yields altogether nine Raman lines. Of these the lines 349  $\text{cm}^{-1}$  and 620  $\text{cm}^{-1}$  are superposed on the halations produced by the over-exposure of the Hg lines 5461 Å and 5770 Å—5791 Å doublet. The other new lines 141  $\text{cm}^{-1}$  and 692  $\text{cm}^{-1}$  are quite sharp. Besides these, the lines 454  $\text{cm}^{-1}$  and 545  $\text{cm}^{-1}$  shift to 442  $\text{cm}^{-1}$  and 536  $\text{cm}^{-1}$  respectively in the solid state. These changes suggest that the molecule undergoes profound changes in the solid state.

The presence of lines having frequency shifts above 545  $\text{cm}^{-1}$  cannot be explained by assuming that the molecule has the form Cl-S-S-Cl. On the contrary, the presence of the line 692  $\text{cm}^{-1}$  in the solid state shows that there is definitely S=S bond in the solid state and the molecule has the configuration  $\text{S}=\text{S}<\overset{\text{Cl}}{\text{Cl}}$ . This molecule also should have yielded only six Raman lines and cannot account for all the nine lines actually ob-

served. The diminution in the frequency of the lines 454 and 545  $\text{cm}^{-1}$  due to S-Cl valence oscillation indicates that some changes occur in this bond. The extra lines might, therefore, be due to the formation of a dimer in the solid state, each of the two molecules having the structure  $\text{S}=\text{S} < \text{Cl}$ .

### Summary

The Raman spectra of diphenyl sulphide, diphenyl methane and sulphur monochloride in the solid and liquid states have been investigated. In the case of the first two compounds in the solid state new lines have been observed in the low frequency region. The frequency shift in the first case is 46  $\text{cm}^{-1}$  and in the second case 50 and 65  $\text{cm}^{-1}$  respectively. In the case of diphenyl sulphide the frequencies of some lines due to C-C vibrations have been observed to diminish in the solid state. In the case of diphenyl methane only three lines 289  $\text{cm}^{-1}$ , 1028  $\text{cm}^{-1}$  and 1188  $\text{cm}^{-1}$  shift slightly when the substance is solidified. In the case of sulphur monochloride the liquid state yields only five lines, but in the solid state nine lines are observed. It is pointed out that the single molecule of  $\text{S}_2\text{Cl}_2$  cannot produce so many

Raman lines and that one of the new lines is due to S=S oscillation, so that in the solid state the structure of the molecule changes from  $\text{Cl.SS}(\text{Cl})_2$  to  $\text{SS}(\text{Cl})_2$  and that the molecules form dimers in the solid state.

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## Preparation & Study of Synthetic Drying Oils

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FOR many years attempts have been made to synthesize oils having good drying properties. The last war gave a great impetus to the development and production of chemically treated, isomerized, reoriented synthetic drying oils<sup>1</sup>.

The shortage of tung oil led to a search for substitutes obtainable by processing available drying oils. Three possibilities suggested themselves: (1) dehydration of castor oil; (2) increasing the effective unsaturation of fatty acids by chemical treatment, fractionation or solvent extraction, and conjuga-

tion of double bonds by treatment with catalysts; and (3) esterification of unsaturated fatty acids by alcohols more polyhydric than glycerol. The last, in particular, had many interesting possibilities.

The method of re-esterification is a natural and logical development based on Carother's theory of functionality<sup>2</sup>, especially as expanded and interpreted by Bradley<sup>3</sup>. In practice, pentaerythritol and higher polymerized alcohols have proved useful for this purpose.

Blagonravova and Drinberg<sup>4</sup> showed that the esterification rates for pentaerythritol

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are essentially the same as those for other polyhydric alcohols, while Burrell<sup>5</sup> showed that the primary hydroxyl groups of pentaerythritol undergo esterification slightly faster than glycerol with its one secondary group. Besides these alcohols sugar alcohols, such as sorbitol and mannitol, can also be employed.

The most widely accepted theory of drying oils is one which is based on the dimensional polymer structure. According to this theory, all polymers which are infusible and insoluble are of the cross linked 3-dimensional form. Natural and synthetic drying oils should possess a structure which will permit the formation of a 3-dimensional polymer.

In order to get a good drying oil from semi- or non-drying oil fatty acids possessing a functionality of 2 (one due to the COOH group, and the other due to unsaturation), the esterification should be carried out with polyhydric alcohols which are more functional than glycerol (which has 3 —OH groups). The greater the number of —OH groups in the alcohol, the better will be the drying and bodying properties of the esters obtained. The introduction of conjugated double bonds during the esterification of the fatty acids will enhance the drying properties. This can be achieved by co-esterifying the given fatty acids with drying oil fatty acids which are rich in conjugated double bonds. The functionality of fatty acids containing conjugated double bonds as a major portion of the total unsaturation may be taken as more than 2 because it is presumed that the Diels-Alder reaction takes place during polymerization.

The advantages to be gained by the presence of conjugated unsaturation in the final esters (oils) can be seen from the results of Lanson and Spoerri's experiments<sup>6</sup>. The polymerization rate of tung-methyl ester is 8—9 times that of soyabean-methyl ester under identical conditions. Further, the copolymerization of one part of tung-methyl ester and two parts of soyabean-methyl ester after 3 hours at 300°C. is 1.8 times the theoretical rate calculated on the basis of individual rates of polymerization of two methyl esters. The probability of conjugated alcostearate radicals reacting with non-conjugated fatty acids by a Diels-Alder type of reaction is assumed.

### Experimental

The present work is divided into 4 parts: In the first part (Series I) cotton seed fatty

acids are esterified with polyhydric alcohols of the type of glycerol, mannitol, sorbitol isomerized mixtures, "Pentek" (a mixture of 85 per cent pentaerythritol and 15 per cent related solid polyhydric alcohol), "Dipentek" (dipentaerythritol alcohol), and "Polypentek" (mixture of polymers of pentaerythritol)\*. The effect of changing the top temperature during esterification on the properties of the final oils were also studied. The second part (Series II) deals with the esterification of "Dipentek" with fatty acids obtained from cotton seed, fish liver oil, fish oil, peanut oil and tall oil. In the third part (Series III) the co-esterification of semi- or non-drying oil fatty acids and drying oil fatty acids is carried out with "Dipentek". In the last part (Series IV) the rate of bodying of the synthetic oil prepared by esterifying cotton seed fatty acids with "Dipentek" is compared with alkali-refined linseed oil with different additives used as accelerators.

The reaction is carried out in a weighed open varnish kettle made of monel metal and having a capacity of about 3-4 gallons. The reaction mixture is stirred at moderate speed. The kettle is weighed after charging, and then put on the fire-grate and heated with a gas burner. The compressed air-driven stirrer is started when the mass has almost melted. The rate of heating is so adjusted that the top temperature is reached in about 60-70 minutes. The first sample is drawn as soon as the top temperature is attained. The temperature is then maintained constant and samples are drawn periodically. After the completion of the reaction, the kettle is cooled, weighed and the product filtered through a strainer. The loss in weight during cooking is calculated by taking the material balance before and after the reaction. The loss is due mainly to the loss of water of esterification and, to a small extent, also due to the escape of volatiles present in the fatty acids.

### Results

Table I shows the formulation and properties of oils produced in the first series of experiments. Figs. 1 and 2 give the rates of esterification. Table II shows the formulation and properties of oils produced in the second series of experiments. Fig. 3 gives the rates of esterification. Table III

\* "Pentek", "Dipentek" and "Polypentek" were obtained from Heyden Chemical Corporation, New York.

TABLE I - SERIES I. FORMULATION &amp; PROPERTIES OF OILS PRODUCED

CODE NUMBER	FORMULATION	% TOTAL CHARGE	TOP TEMP. °F.	% LOSS IN COOKING	BODY*	REF. INDEX AT 55° F.	PROPERTIES OF PROCESSED OIL			REMARKS
							Gelling time at 530°F. or body after 6 hr. at 530°F.	Drying of film of 1.5 mil thickness with 0.24 Ca as Ca-naphthenate at room temp.	Acid No.	
Co-V	Cotton seed fatty acids	89.0	450	15.5	Semi-solid	...	...	...	...	...
Co-VI	Glycerol	11.0								
	Cotton seed fatty acids	83.8	450	12.6	½hH	1.4808	U	Tacky after 12 days	11.2	...
	Sorbitol isomerized mix.	16.2								
Co-VIII	Cotton seed fatty acids	84.6	450	14.7	F	1.4809	V	Very tacky aft. 12 days	23.5	...
	Mannitol	15.4								
Co-IV	Cotton seed fatty acids	87.0	450	10.4	½ thin	1.4785	½hW	"	10.5	...
	" Pentek "	13.0								
Co-III	Cotton seed fatty acids	85.0	450	11.3	½hJ	1.4790	½ thin Y	"	11.7	...
	" Dipentek "	15.0								
Co-VII	Cotton seed fatty acids	84.0	450	7.5	½hI	1.4815	X	Tacky aft. 12 days	12.5	8.3% gel
	" Polypentek "	16.0								
Co-X	Cotton seed fatty acids	87.0	450	7.4	½hE	1.4795	½ thin U	Tacky aft. 8 days	11.5	...
	" Pentek "	13.0								
	Ca-naphthenate	0.012								
Co-XI	Cotton seed fatty acids	87.0	500	11.6	½hF	1.4800	½hT	Slightly tacky aft. 8 days	3.8	...
	" Pentek "	13.0								
Co-XII	Cotton seed fatty acids	85.0	500	12.4	½hI	1.4815	Y	Very slightly tacky aft. 8 days	11.0	...
	" Dipentek "	15.0								
Co-XIII	Cotton seed fatty acids	84.0	500	11.8	K	1.4815	Y	Slightly tacky aft. 8 days	16.0	...
	" Polypentek "	16.0								

\*Gardner-Holdt Scale, see Gardner, H.A., *Physical & Chemical Examination of Paints, Varnishes, Lacquers & Colors* (Institute of Paint & Varnish Research, Washington D.C.), 1939, 9th ed.

TABLE II - SERIES II. FORMULATION &amp; PROPERTIES OF OILS PRODUCED

CODE NUMBER	FORMULATION	% TOTAL CHARGE	TOP TEMP. °F.	% LOSS IN COOKING	BODY*	REF. INDEX AT 55° F.	PROPERTIES OF PROCESSED OIL			REMARKS
							Gelling time at 530° F. or body after 6 hr. at 530° F.	Drying of film of 1.5 mil thickness with 0.24% Co as Co-naphthenate at room temp.	Acid No.	
C-4	Cotton seed fatty acids	86.3	500	12.0	½hI	1.4780	½ thin Y	Very slightly tacky aft. 12 days	9.5	...
	" Dipentek "	13.7								
C-5	Fish liver fatty acids	88.0	500	13.7	½hT	1.4890	Z-6	Very slightly tacky aft. 5 days; tack free aft. 12 days	6.7	...
	" Dipentek "	12.0								
C-9	Tall oil	87.2	500	12.0	1 bubble thin A	1.4690	...	Tack free aft. 1 day	15.8	Diluted to 50% solid with benzene
	" Dipentek "	12.8								
P-4	Peanut fatty acids	86.3	500	12.5	J	1.4790	...	Tacky aft. 9 days	9.2	...
	" Dipentek "	13.7								
F-1	Fish oil fatty acids	87.6	500	11.4	¾ thin U	1.4878	350 min.	Tack free aft. 1 day	9.2	...
	" Dipentek "	12.4								

\*Gardner-Holdt Scale.

shows the formulation and properties of oils produced in the third series of experiments. Figs. 4-8 give the rates of esterification. Table IV shows the formulation and properties of oils produced in the fourth series of experiments and indicates the rates of bodying when various additives are employed as accelerators.

## Discussion

The tables and graphs give a picture of the rates of esterification, and the drying properties of products obtained by esterifying cotton seed fatty acids with glycerol, mannitol, sorbitol isomerized mixture, "Pentek", "Dipentek", and "Polypentek". Considering the penta series of alcohols, it will

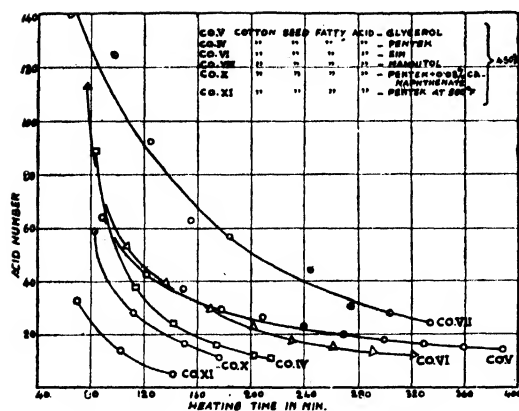


FIG. 1 — RATE OF ESTERIFICATION.

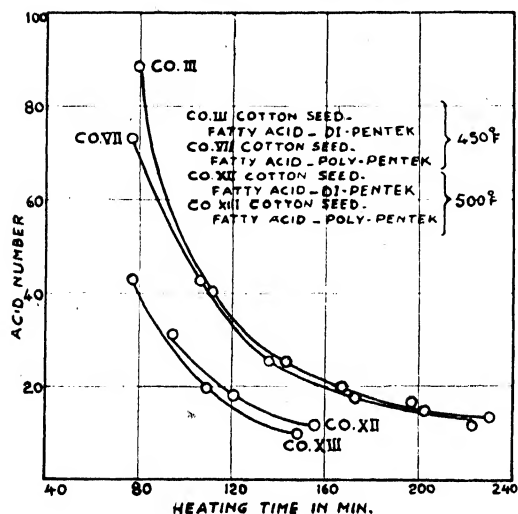


FIG. 2 — RATE OF ESTERIFICATION.

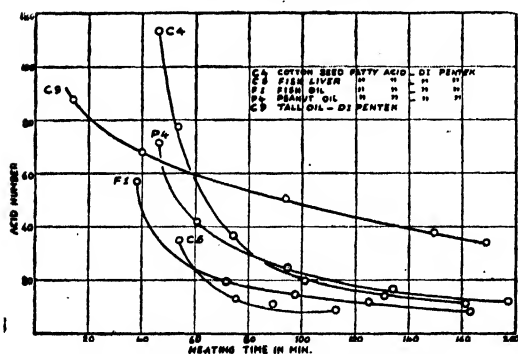


FIG. 3 — RATE OF ESTERIFICATION.

be seen that esterification of the fatty acids with "Polypentek" is faster than that with "Dipentek", which in turn is faster than that with "Pentek". The rate of esterification increases with temperature.

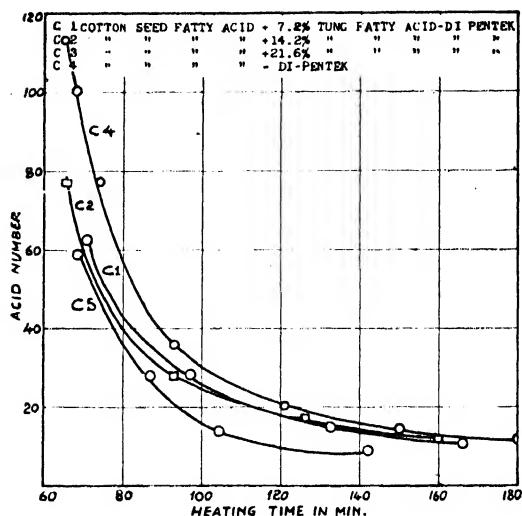


FIG. 4 — RATE OF ESTERIFICATION.

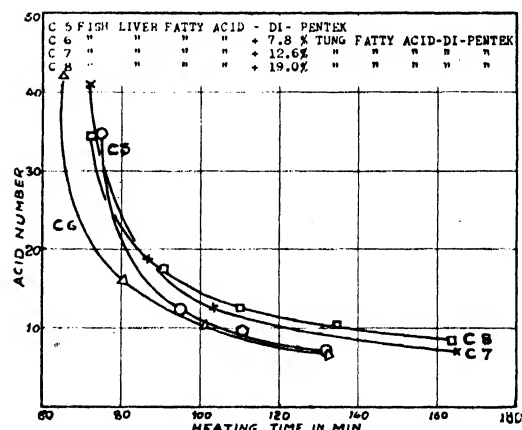


FIG. 5 — RATE OF ESTERIFICATION.

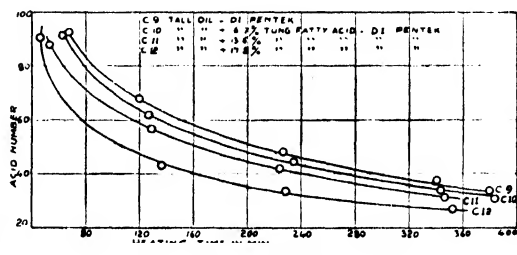


FIG. 6 — RATE OF ESTERIFICATION.

The use of 0.08 per cent calcium (as Ca-naphthenate) on the weight of "Pentek" enhances both the rate of esterification of cotton seed fatty acids, and the rate of drying of the final product.

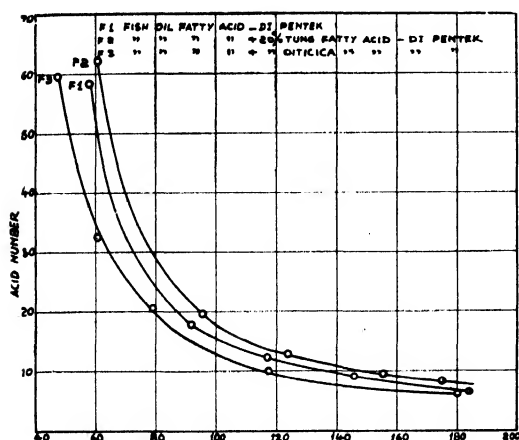


FIG. 7 -- RATE OF ESTERIFICATION.

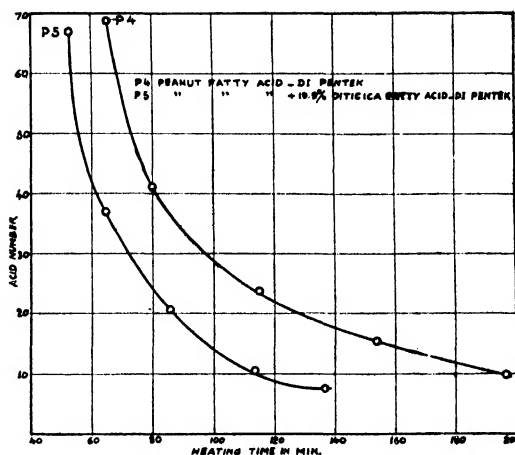


FIG. 8 -- RATE OF ESTERIFICATION.

TABLE III -- SERIES III. FORMULATION &amp; PROPERTIES OF OILS PRODUCED

CODE NUMBER	FORMULATION	% TOTAL CHARGE	TOP TEMP. °F	BODY*	% LOSS IN COOKING	REF. INDEX AT 72°F.	PROPERTIES OF PROCESSED OIL			REMARKS
							Gelling time at 530°F. or body after 6 hr. at 530°F.	Drying of film of 1.5 mil thickness with 0.24% Co as Co-naphthenate at room temp.	Acid No.	
C-1	Cotton seed fatty acids	79.0	500	R	10.8	1.4815	1 thin Z-4	Tack free aft. 29 hr.	10.1	
	Tung fatty acids	20.2								
	"Dipentek"	13.8								
C-2	Cotton seed fatty acids	72.0	500	1hV	11.1	1.4840	Almost solid	Very slightly tacky aft. 6 hr. and tack free aft. 1 day	11.2	...
	Tung fatty acids	14.2								
	"Dipentek"	13.8								
C-3	Cotton seed fatty acids	64.6	500	1 thin Z-6	11.5	1.4880	85 min.	Very slightly tacky aft. 6 hr. and tack free aft. 1 day	8.0	...
	Tung fatty acids	21.6								
	"Dipentek"	13.8								
C-6	Fish liver fatty acids	80.2	500	W-1	15.1	1.4912	Z-6	Slightly tacky aft. 1 day and tack free aft. 8 days	6.7	...
	Tung fatty acids	7.8								
	"Dipentek"	12.2								
C-7	Fish liver fatty acids	75.2	500	1hZ-1	22.4	1.4932	250 min.	Very slightly tacky aft. 1 day and tack free aft. 5 days	6.7	...
	Tung fatty acids	12.6								
	"Dipentek"	12.2								
C-8	Fish liver fatty acids	68.7	500	Heavier than Z-6	21.5	1.4950	80 min.	Tack free aft. 1 day	8.3	...
	Tung fatty acids	19.0								
	"Dipentek"	12.3								
C-10	Tall oil	80.3	500	thin A	13.6	1.4691	...	Tack free aft. 1 day	14.6	Diluted to 50% solids with benzene
	Tung fatty acids	6.7								
	"Dipentek"	13.0								
C-11	Tall oil	73.6	500	thin A	11.7	1.4692	...	Tack free aft. 1 day	15.0	do
	Tung fatty acids	13.4								
	"Dipentek"	13.0								
C-12	Tall oil	69.5	500	C	...	1.4705	...	Tack free aft. 1 day	12.6	do
	Tung fatty acids	17.5								
	"Dipentek"	13.0								
F-2	Fish oil fatty acids	72.8	500	2 1/2 h Z-6	10.4	1.4920	0.0 min. (before top temp. is reached)	Very slightly tacky aft. 1 day and tack free aft. 2 days	7.8	...
	Tung fatty acids	14.6								
	"Dipentek"	12.6								
F-3	Fish oil fatty acids	72.0	500	1 h Z-4	11.3	1.4920	100 min.	Tack free aft. 7 hr.	5.8	...
	Oiticica fatty acids	15.0								
	"Dipentek"	13.0								
P-5	Peanut fatty acids	96.6	500	1 h Z-4	17.2	1.4910	...	...	7.8	...
	Oiticica fatty acids	19.9								
	"Dipentek"	13.7								

\*Gardner-Holdt Scale.

In the case of "Polypentek"-cotton seed fatty acids esters, a certain degree of gel formation is noticed. This is attributed to

the impurities in "Polypentek" (technical grade) used for the esterification and also to the very complex structure of the alcohol.

TABLE IV — CHANGES IN BODYING, REF. INDEX &amp; ACID NUMBER OF SYNTHETIC OIL CO-XII, &amp; ALKALI-REFINED LINSEED OIL WITH DIFFERENT ADDITIVES

	SAMPLE No						
	1	2	3	4	5	6	7
<b>1. Cov-I — 95.5 parts Co-XII oil &amp; 4.5 parts maleic anhydride</b>							
Hr. after top temp.	0.0	1.0	2.0	3.0	4.0	5.0	6.0
Body in poises	5.7	10.7	12.9	16.0	22.7	30.2	54.3
Acid No.	18.4	13.0	10.0	7.75	7.0	6.25	5.2
Ref. index at 50°F.	1.4830	1.4845	1.4852	1.4855	1.4860	1.4862	1.4865
<b>2. Cov-II — 95.5 parts linseed oil &amp; 4.5 parts maleic anhydride</b>							
Hr. after top temp.	0.0	1.0	2.0	3.0	4.0	5.0	6.0
Body in poises	0.85	1.65	2.75	4.85	9.32	19.2	41.2
Acid No.	15.5	6.25	6.75	8.75	9.25	9.0	7.75
Ref. index at 55°F.	1.4870	1.4884	1.4898	1.4910	1.4922	1.4931	1.4944
<b>3. Cov-III — 95.5 Co-XII oil &amp; 4.5 parts crotonic acid</b>							
Hr. after top temp.	0.0	1.0	2.0	3.0	4.0	5.0	6.0
Body in poises	2.44	3.05	3.85	5.5	7.55	9.7	15.2
Acid No.	2.5	2.25	2.5	3.25	2.5	2.75	3.25
Ref. index at 64°F.	1.4800	1.4802	1.4804	1.4813	1.4819	1.4822	1.4828
<b>4. Cov-IV — 95.5 linseed oil &amp; 4.5 parts crotonic acid</b>							
Hr. after top temp.	0.0	1.0	2.0	3.0	4.0	5.0	6.0
Body in poises	0.45	0.65	0.85	1.0	1.65	2.13	3.3
Acid No.	6.0	2.5	1.75	1.5	1.25	1.25	1.25
Ref. index at 70°F.	1.4819	1.4822	1.4830	1.4836	1.4845	1.4849	1.4850
<b>5. Cov-V — 95.5 parts Co-XII oil &amp; 4.5 parts sorbic acid</b>							
Hr. after top temp.	0.0	1.0	2.0	3.0	4.0	5.0	6.0
Body in poises	2.92	3.85	4.7	6.27	8.86	9.7	15.3
Acid No.	11.0	5.5	3.0	2.0	1.75	1.5	1.25
Ref. index at 73°F.	1.4778	1.4787	1.4798	1.4802	1.4810	1.4815	1.4820
<b>6. Cov-VI — 95.5 parts linseed oil &amp; 4.5 parts sorbic acid</b>							
Hr. after top temp.	0.0	1.0	2.0	3.0	4.0	5.0	6.0
Body in poises	0.58	0.75	1.0	1.4	2.25	3.15	4.18
Acid No.	4.25	5.0	4.75	4.0	3.5	3.5	3.25
Ref. index at 70°F.	1.4820	1.4832	1.4840	1.4848	1.4856	1.4865	1.4872

REMARKS: Films of 1.5 mil thickness, and 0.24% Co as Co-naphthenate tack free at room temperature after: (1) 4 days; (2) 4 days; (3) 7 days; (4) 4 days; (5) 7 days; (6) 4 days.

Comparing the properties of esters obtained by the use of alcohols other than those of the "Pentek" series, it will be noticed that the product obtained with sorbitol isomerized mixture shows a good drying rate.

The drying rates of the esters are improved as the temperature of esterification is increased. At a temperature of 450°F. "Polypentek" ester dries better than "Pentek" or "Dipentek" esters. The "Pentek" ester prepared at a top temperature of 500°F. is comparable in drying properties to the "Polypentek" ester prepared at a top temperature of 450°F.

The rate of bodying of oils increases in the order: sorbitol isomerized mixture, mannitol, "Pentek", "Dipentek" and "Polypentek" esters.

The fish liver oil fatty acids are the fastest, and the tall oil the slowest, to esterify with "Dipentek" as judged by acid numbers. Fish oil fatty acids, cotton seed oil fatty acids and peanut oil fatty acids come in

between the fish liver oil fatty acids and tall oil acids.\*

The refractive indices of the esters decrease in the order, fish liver oil, fish oil, peanut and cotton seed fatty acids.

The drying rate of fish oil fatty acids esters is high, that of the peanut fatty acids esters being the lowest. The cotton seed fatty acids esters dry better than fish liver fatty acids esters, but the film obtained is tacky even after 12 days. Cotton seed, fish liver and fish oil fatty acids esters progressively show an increasing rate of bodying.

The use of tung oil fatty acids in co-esterification enhances the drying rate, the rate of bodying and the refractive index of the fatty acids esters. The higher the percentage of tung oil fatty acids in the mixture, the higher is the improvement in

\*Since tall oil (Union Bag & Paper Corporation, New York; fatty acids 48-52 per cent and rosin acids 42-46 per cent) ester is a semi-solid due to the presence of abietic acid ester, a 50 per cent solution in benzene is used for evaluating its properties.

the drying rate, bodying rate and refractive index. The addition of oiticica oil fatty acids also improves the drying rate and the rate of bodying. These improvements are due to a net increase in the amount of total as well as conjugate types of unsaturation per molecule of ester formed.

The rate of bodying of the synthetic oil CO-XII is compared with the bodying rate of alkali-refined linseed oil in the presence of different additives. The synthetic oil bodies faster than the alkali-refined linseed oil, but the refractive index of bodied linseed oil is higher than that of the synthetic oil CO-XII. The drying rates of bodied linseed oils are superior to those of the corresponding bodied synthetic oil.

The fast rate of bodying (heat polymerization) of the synthetic oil compared to linseed oil may be explained on the basis that the polyhydric alcohol "Dipentek" used in the former case is much more complex and polyhydric in nature than glycerol which is present in linseed.

The fast drying rate (oxygen polymerization) of the boiled linseed oil indicates that

(a) the total amount of unsaturation per molecule of bodied linseed oil is more than that present in a molecule of the bodied synthetic oil; and/or (b) a part of the total unsaturation of the linseed oil molecule is of the conjugated type. In so far as oxygen polymerization is concerned, conjugate unsaturation is considered to be more effective than an equivalent amount of unsaturation due to isolated double bonds as the former gives Diels-Alder reaction.

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## Carotenoid Pigments of Common Indian Fruits

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**I**N earlier papers, the results of investigations on the carotenoid pigments of different varieties of maize<sup>1</sup>, mango<sup>2</sup>, carrots<sup>3</sup>, oranges<sup>4</sup>, and common vegetables<sup>5</sup> have been reported. The results relating to the carotenoid pigments of some common Indian fruits such as water-melon, peach, mango, papaya, apricot, pine-apple, banana, guava, musk-melon and Mosambique oranges are reported in this paper.

The carotenoid pigments of fruits are varied, and isomers of the same pigment are often present. Observations on the total hydrocarbon pigments of fruits have been reported by earlier workers<sup>6-14</sup>.

All the fruits were obtained from the market, and were of the quality commonly

consumed. The procedure followed for the complete extraction, isolation and estimation of the different carotenoid pigments has been described in an earlier paper<sup>4</sup>. The quantities of different pigments were estimated colorimetrically by referring to a standard curve of  $\beta$ -carotene against 0.04 per cent potassium dichromate. The same curve was used for the estimation of all the pigments except for lycopene and neo-lycopene. In the case of these two pigments, the solutions of the pigments were matched against 0.001 per cent methyl orange and the quantities read off from a standard curve showing the relation between the colour intensities of different concentrations of pure lycopene and 0.001 per cent methyl orange.

The results are expressed as  $\mu\text{gm.}/\text{gm.}$  of the fresh material.

On the basis of the biological activity of the various carotenoid pigments reported in the literature, the vitamin A potency is calculated according to the following formula :

Vitamin A potency in I.U. per gm.

$$\begin{aligned}
 &= \frac{\mu\text{gm. } \beta\text{-carotene}}{0.6} \\
 &+ \frac{\mu\text{gm. } \alpha\text{-carotene}}{1.2} \quad (\text{Wilkinson}^{15}) \\
 &+ \frac{\mu\text{gm. } \gamma\text{-carotene}}{1.2} \quad (\text{Kuhn, et. al.}^{16}) \\
 &+ \frac{\mu\text{gm. neo-}\beta\text{-carotene B}}{1.2} \quad (\text{Deuel, et. al.}^{17}) \\
 &+ \frac{\mu\text{gm. neo-}\beta\text{-carotene U}}{2.4} \quad (\text{Kemmerer and Fraps}^{18}) \\
 &+ \frac{\mu\text{gm. kryptoxanthin}}{1.2} \quad (\text{Deuel, et. al.}^{19}) \\
 &+ \frac{\mu\text{gm. neo-kryptoxanthin}}{1.2} \quad (\text{Fraps and Kemmerer}^{20})
 \end{aligned}$$

The analytical data on the carotenoid pigments present in the various fruits are given in Tables I to III. Table I gives the results of analyses of fruits rich in carotenoids, viz. water-melon, peach and mango ; Table II of fruits moderately rich in carotenoids, viz. papaya and apricot ; and Table III of fruits poor in carotenoids.

### Discussion

*Water-melon* (*Citrullus vulgaris*) — Lycopene and carotene from the fruit were isolated by Zechmeister and Tuzson<sup>21</sup> who showed that lycopene, the chief pigment, was responsible for its red colour. Zechmeister and Polgar<sup>22</sup> detected 6 different carotenoid pigments in this fruit, viz. xanthophyll, lycopene,  $\gamma$ -carotene,  $\beta$ -carotene and  $\alpha$ -carotene and an unknown carotenoid which is located between  $\gamma$ -carotene and  $\beta$ -carotene in the adsorption column. In our studies we have not been able to detect  $\gamma$ -carotene and the unknown pigment which is adsorbed on the column between  $\gamma$ -carotene and  $\beta$ -carotene in Zechmeister and Polgar's<sup>22</sup> investigations, but an additional unidentified band which is adsorbed just below xanthophyll was detected.

A typical chromatogram is shown in Fig. 1.

*Peach* (*Amygdalus persica*) — Strain<sup>25</sup> and Mackinney<sup>26</sup> using magnesium oxide as an adsorbant found the carotenoids in peaches to consist of  $\beta$ -carotene, 35-40 per cent ; kryptoxanthin, 25-30 per cent ; and the remainder to be lutein and zeaxanthin. Thaler, et al<sup>27</sup>, showed that the meat from

yellow peaches contains lutein,  $\beta$ -carotene and lycopene in the ratio 8 : 2 : 1 ; Kemmerer and Fraps<sup>28</sup> reported that peaches do not contain measurable quantities of lycopene. It cannot occur to the extent of 9-10 per cent as indicated. In our studies, 5 pigments were isolated from peach, viz.

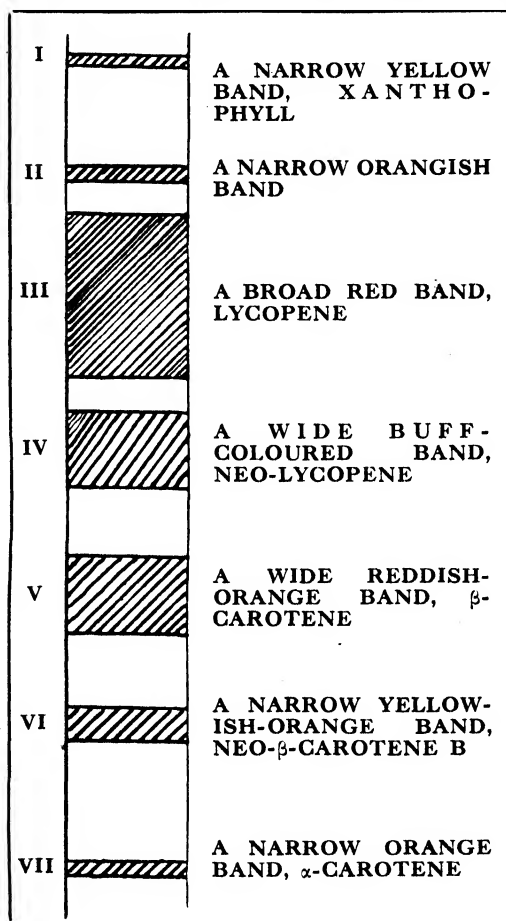


FIG. 1—TYPICAL CHROMATOGRAM OF THE PIGMENTS OF WATER-MELON.

- BAND I REPRESENTS XANTHOPHYLL AS SHOWN BY PHASE SEPARATION.
- BAND II IS DUE TO A PIGMENT WHICH HAS NOT BEEN IDENTIFIED.
- BAND III REPRESENTS LYCOPENE AS SHOWN BY ITS ABSORPTION SPECTRUM.
- BAND IV IS PRESUMABLY AN ISOMER OF LYCOPENE, CALLED NEO-LYCOPENE BY ZECHMEISTER AND TUZSON<sup>21</sup>.
- BAND V REPRESENTS  $\beta$ -CAROTENE AS SHOWN BY ITS ABSORPTION SPECTRUM.
- BAND VI IS AN ISOMER OF  $\beta$ -CAROTENE CALLED NEO- $\beta$ -CAROTENE B BY POLGAR AND ZECHMEISTER<sup>22</sup>.
- BAND VII HAS BEEN IDENTIFIED AS  $\alpha$ -CAROTENE BY ITS ABSORPTION SPECTRUM.

**TABLE I — RICH SOURCES OF CAROTENOID PIGMENTS**

FRUIT AND SOURCE OF THE SAMPLE	XANTHO- PHYLL		NEO- XANTHO- PHYLL		UNIDENTI- FIED PIGMENT		KRYPTO- XANTHIN		LYCOPENE		NEO- LYCOPENE		NEO- $\beta$ - CAROTENE U		$\beta$ -CAROTENE		NEO- $\beta$ - CAROTENE B		$\alpha$ -CAROTENE		TOTAL ACTIVE PIGMENTS AS $\beta$ -CAROTENE		VITAMIN A POTENCY
	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%	$\mu\text{gm./}$ gm.	%			
Water-melon ( <i>Citrullus vulgaris</i> )																							
Delhi	34.2	0.3	0.9	...	0.2	0.6	...	...	33.6	98.2	...	14.9	19.7	...	0.1	0.3	...	...	...	...	0.1	0.3	0.2
Gulshar	75.4	0.4	0.5	...	0.2	0.3	...	...	59.6	79.0	...	14.9	19.7	...	0.3	0.4	...	...	...	...	0.3	0.4	0.5
Gulshar	138.6	0.8	0.6	...	1.0	0.7	...	...	103.3	74.4	...	27.6	19.9	...	5.0	3.6	...	0.6	0.2	0.1	5.5	4.0	9.2
Farrukhabad	140.7	0.6	0.4	...	1.1	0.8	...	...	108.3	76.9	...	26.4	18.7	...	3.3	2.3	...	0.9	0.6	0.1	3.8	2.7	6.3
Peach ( <i>Amygdalus persica</i> )																							
Delhi, unripe	2.3	1.5	65.2	...	...	...	...	0.2	8.7	...	...	...	...	...	0.3	13.0	...	4.3	...	...	0.45	19.6	0.8
" ripe	20.0	16.0	80.0	...	...	...	1.1	5.5	0.6	3.0	...	...	...	...	2.0	10.0	...	1.5	...	...	2.70	13.5	4.5
Quetta	68.3	56.0	81.9	...	...	...	5.6	8.2	0.9	1.3	...	...	...	...	4.9	7.1	...	0.9	1.3	...	8.15	11.9	13.6
Peshawar	78.3	62.5	79.8	...	...	...	3.9	7.5	2.4	3.1	...	...	...	...	6.5	8.3	...	1.0	1.2	...	9.95	12.7	16.6
Mango ( <i>Mangifera indica</i> )																							
Bombay, Perry	62.8	31.5	50.2	...	...	...	...	...	...	...	...	...	...	...	3.1	5.0	...	4.3	6.8	...	26.82	42.7	44.7
" Tinda	13.7	2.9	21.2	...	...	...	...	...	...	...	...	...	...	...	0.5	3.6	...	2.1	15.3	...	9.37	68.4	15.6
" Alphonso	135.6	61.2	45.1	3.5	2.6	...	...	...	...	...	...	...	...	...	2.4	51.6	...	38.0	16.9	...	60.65	44.7	101.1
" Safaidda	17.3	5.9	34.1	0.4	2.3	...	...	...	...	...	...	...	...	...	0.4	2.3	...	52.6	1.5	...	9.95	57.5	16.6
" Surkha	14.9	7.5	50.3	...	...	...	...	...	...	...	...	...	...	...	0.5	3.8	...	41.6	0.7	...	6.67	44.8	11.1
" Totla Pari	21.9	10.4	47.5	...	...	...	...	...	...	...	...	...	...	...	0.3	1.3	...	10.0	45.6	...	10.67	48.7	17.8
" Malda	10.0	4.9	49.0	...	...	...	...	...	...	...	...	...	...	...	0.3	3.0	...	37.0	1.1	...	4.32	43.2	7.2
Sabarapur,																							
Ramkaila	17.5	8.5	48.5	...	...	...	...	...	...	...	...	...	...	...	1.0	5.7	...	40.0	1.0	...	7.75	44.3	12.9
Saroli	164.9	42.1	25.5	...	...	...	...	...	...	...	...	...	...	...	7.3	4.4	...	96.3	56.4	...	107.72	65.3	179.5

TABLE II — MODERATELY RICH SOURCES OF CAROTENOID PIGMENTS

FRUIT AND SOURCE OF THE SAMPLE	TOTAL ZEAXANTHIN		XANTHOPHYLL		CRYPTO- XANTHIN		NEO-KRYPTO- XANTHIN		LYCOPENE		NEO-β- CAROTENE U		β-CAROTENE		NEO-β- CAROTENE B		TOTAL ACTIVE PIGMENTS AS β-CAROTENE		VITAMIN A POTENCY I.U./gm.	
	μgm./ gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%		
Papaya ( <i>Carica papaya</i> ) unripe	3.9	2.3	59.0	...	...	...	1.2	30.8	...	...	...	...	...	0.3	7.7	0.1	2.6	0.95	24.4	1.6
" light yellow,	5.6	2.2	39.3	...	...	...	2.9	51.8	...	...	...	...	...	0.4	7.1	0.1	0.8	1.90	33.9	3.2
" light yellow,	9.2	3.8	41.3	...	...	...	4.9	53.2	...	...	...	...	...	0.4	4.3	0.1	1.1	2.90	31.5	4.8
" deep yellow,	19.99	4.1	20.5	...	...	...	11.7	58.5	0.29	1.5	...	...	...	3.1	15.5	0.8	4.0	9.49	47.5	15.8
" ripe	21.9	4.8	22.0	...	...	...	13.0	59.3	0.3	1.4	...	...	...	3.3	15.0	0.5	2.3	10.20	46.5	17.0
Apricot ( <i>Prunus armeniaca</i> ) unripe	2.7	...	...	1.0	37.0	...	...	...	...	0.4	14.8	...	...	1.1	40.7	0.2	7.4	1.2	44.4	2.0
" Kashmir, light yellow	15.7	...	...	1.9	23.2	...	...	...	...	1.0	12.2	0.2	2.4	4.6	56.1	0.5	6.1	4.9	59.2	8.2
" Smila, orange	...	...	...	3.2	20.4	...	...	...	...	1.6	10.2	0.9	5.7	8.6	54.8	1.4	8.9	9.5	60.5	15.8



TABLE III — POOR SOURCES OF CAROTENOID PIGMENTS

FRUIT AND SOURCE OF THE SAMPLE	TOTAL PIGMENTS	XANTHOPHYLL		NEO-XANTHOPHYLL		NEO- $\beta$ -CAROTENE U		$\beta$ -CAROTENE		NEO- $\beta$ -CAROTENE B		TOTAL ACTIVE PIGMENTS AS $\beta$ -CAROTENE		VITAMIN A POTENCY I.U./gm.
	$\mu\text{gm./gm.}$	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	
Pine-apple ( <i>Ananas sativus</i> )														
Assam	1.9	1.3	68.5	...	...	0.1	5.2	0.4	21.0	0.1	5.2	0.47	22.9	0.8
Singapore	7.7	5.7	74.0	0.3	3.9	0.2	2.6	1.2	15.6	0.3	3.9	1.40	18.2	2.3
Banana ( <i>Musa sapientum</i> )														
Galleon (Bombay), red	5.1	2.2	43.1	...	...	0.3	5.9	2.6	51.0	...	...	2.67	52.3	4.5
Basin (Bombay), chitri	1.7	1.0	58.8	...	...	0.1	5.8	0.6	35.3	...	...	0.62	36.5	1.0
Guava ( <i>Psidium guajava</i> )														
Allahabad, pulp	0.2	0.1	50.0	...	...	...	...	0.1	50.0	...	...	0.1	50.0	0.2
" peel	1.9	1.2	63.1	...	...	...	...	0.7	36.9	...	...	0.7	36.9	1.2
" peel	2.1	1.5	71.4	...	...	...	...	0.6	28.6	...	...	0.6	28.6	1.0
Musk-melon or Kabul-melon ( <i>Cucumis melo</i> )														
Quetta	0.2	0.1	50.0	...	...	...	...	0.1	50.0	...	...	0.1	50.0	0.2
"	0.8	0.3	37.5	...	...	...	...	0.5	62.5	...	...	0.5	62.5	0.8
Grapes ( <i>Vitis vinifera</i> )														
Peshawar	1.6	1.1	68.7	...	...	...	...	0.5	31.2	...	...	0.5	31.2	0.8
Chaman	2.1	1.4	66.6	...	...	...	...	0.7	33.3	...	...	0.7	33.3	1.1
Mosambique oranges														
Bombay, thick rind, juicy	2.8	2.3	82.1	...	...	...	...	0.4	14.3	0.1	3.6	0.45	16.0	0.8
" " not juicy	3.1	2.4	77.4	...	...	...	...	0.5	16.1	0.2	6.4	0.60	19.3	1.0
" " thin rind, juicy	2.9	2.4	82.7	...	...	...	...	0.4	13.8	0.1	3.5	0.45	15.5	0.75

xanthophyll, kryptoxanthin, lycopene,  $\beta$ -carotene and neo- $\beta$ -carotene B.

**Mango (*Mangifera indica*)**—The carotenoids of 10 different varieties of mangoes have already been reported<sup>2</sup>. 10 more varieties have been examined. 5 different pigments were isolated from the various varieties examined. These are xanthophyll, neo-xanthophyll,  $\beta$ -carotene, neo- $\beta$ -carotene U and neo- $\beta$ -carotene B.

**Papaya (*Carica papaya*)**—In 1933 Yamamoto and Tin<sup>29</sup> discovered a new pigment in the fruit of the papaya to which they gave the name "caricaxanthin" and which they found was similar to the pigment found in oranges. Karrer and Schlienz<sup>30</sup> pointed out that caricaxanthin of Yamamoto and Tin was similar to kryptoxanthin as both these pigments had the same absorption spectrum and melting point. Karrer and Schlienz<sup>30</sup> isolated zeaxanthin, kryptoxanthin, and  $\beta$ -carotene from the papaya fruit. In our studies 5 different pigments were isolated and identified. These include zeaxanthin, kryptoxanthin,  $\beta$ -carotene, neo- $\beta$ -carotene B and neo- $\beta$ -carotene U.

**Apricot (*Prunus armeniaca*)**—Morgan and Madsen<sup>7</sup> reported that xanthophyll was absent in apricots as the methyl alcohol washings were colourless. Brockmann<sup>31</sup> isolated from dried apricots,  $\beta$ -carotene in crystalline form and lycopene in small amounts, and also reported the presence of small amounts of  $\gamma$ -carotene. Mackinney, Aronoff and Bornstein<sup>32</sup> reported that xanthophyll, lycopene and  $\gamma$ -carotene do not

account for 10 per cent of the total carotenoids. Kemmerer and Fraps<sup>33</sup> demonstrated the presence of 4 different pigments in canned apricots. These are  $\beta$ -carotene, 56.1 per cent; neo- $\beta$ -carotene U, 5.9 per cent; neo- $\beta$ -carotene B, 7.2 per cent; and an impurity A to the extent of 3.8 per cent. In our studies 5 different pigments were isolated and identified. These include xanthophyll, lycopene, neo- $\beta$ -carotene U,  $\beta$ -carotene, and neo- $\beta$ -carotene B.

**Pine-apple (*Ananas sativus*)**—Pine-apple fruit owes its yellow colour to carotene and xanthophyll. Magistard<sup>34</sup> reported that in the pine-apple fruit xanthophyll was present to the extent of 2.7  $\mu\text{gm./gm.}$  and carotene to the extent of 2.8  $\mu\text{gm./gm.}$  Two varieties of pine-apples grown in India were studied for their carotenoid content. 5 different pigments were isolated, namely xanthophyll, neo-xanthophyll,  $\beta$ -carotene, neo- $\beta$ -carotene U and neo- $\beta$ -carotene B.

**Banana (*Musa sapientum*) ; Guava (*Psidium guajava*) ; Musk-melon (*Cucumis melo*) ; Grapes (*Vitis vinifera*) & Mosambique Oranges**—The amount of carotenoid pigments present in these fruits is very small. 2 different pigments were isolated from all the fruits except in the case of Mosambique oranges and banana. In the former neo- $\beta$ -carotene B and in the latter neo- $\beta$ -carotene U were also detected in addition to xanthophyll and  $\beta$ -carotene. In the case of guava the carotenoid pigments present both in the peel and the pulp were determined separately. The guava peel has been found to be

richer both in carotene and xanthophyll than the pulp.

### Summary

The carotenoid pigments present in different varieties of 11 common Indian fruits have been isolated, identified and estimated by the chromatographic technique. The fruits studied include water-melon, peach, mango, papaya, apricot, pine-apple, banana, guava, musk-melon, grapes and Mosambique oranges.

Out of the fruits examined, mango has been found to be a rich source of vitamin A, while peach, papaya and apricot are moderately rich sources of the vitamin. Water-melon, though rich in total carotenoids, is poor in the provitamin A. Banana, pine-apple, grapes, guava, musk-melon and Mosambique oranges are poor sources of vitamin A.

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## Grain-size Properties of Some Railway Steels

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IT has now been universally established that austenitic grain-size control of steel confers upon it a set of characteristic beneficial properties. The theories and mechanisms of austenitic grain-size control have been discussed by the author in an earlier paper<sup>1</sup>. Briefly, austenitic grain

size of steels is influenced by the deoxidation practice of steel making in general and by aluminium additions in particular. Strong deoxidation with aluminium renders the steel fine grain, while a steel containing little or no aluminium is coarse grained — its austenitic grains coarsen rapidly at high

temperatures. The aluminium content of coarse grained steels varies up to 0.003 per cent. Normally an excess of 0.02—0.04 per cent aluminium over that required for de-oxidation makes the steel fine grained. Railway steels made two decades back were mostly coarse grained.

It is with the railway steels of past days, and with some of those of recent make, partially or fully deoxidized with aluminium, that this investigation is concerned. The railway steels made twenty to thirty years ago were taken for investigation after the products made from them, e.g. tyre, wheels, etc., had worn down too thin to be safely employed. The old tyre steels examined had mostly done 300,000 to 400,000 miles of actual service mileage under heavy traffic loads, whereas the new steels put up for relative study had yet to enter service. These new steels contained up to a maximum of 0.015 per cent aluminium in solution.

Several factors determine the amount of aluminium added to steel, e.g. the carbon content, temperature, state of deoxidation, silicon content of the bath, the manner of adding aluminium and the form of aluminium used. Generally, the lower the carbon content, the greater is the amount of aluminium required for grain-size control. Less aluminium will be required for a steel of 0.25—0.30 per cent silicon than one of 0.15—0.20 per cent silicon. The state of deoxidation of the bath at tap is indicated by the residual silicon content.

#### Coarse & Fine Grained Steels

Inherently fine grained steels possess certain marked favourable physical properties compared to the inherently coarse grained type, the most outstanding of which is their high impact toughness.

**Impact & Tensile Properties** — In normalized condition the fine grained steels have a slightly lower ultimate tensile strength, a higher elongation and a substantially higher notched-bar toughness. This is illustrated in a 0.40 per cent carbon steel as follows :

Grain size	Tensile strength tons/sq. in.	Elongation	Izod impact ft. lb.
Coarse	44	22	15
Fine	40	28	55

The author obtained the following values with 0.20 per cent carbon steels, one treated with aluminium and the other aluminium free, both in the normalized state :

Fine grain	.. 82.4 ft. lb. average.
Coarse grain	.. 37.6 ft. lb. average.

When inherently fine and coarse grained specimens of the same steel are quenched from above the critical temperature range and tempered under the same conditions, the former have a slightly lower tensile strength, a slightly higher elongation and reduction in area and a substantially higher notched-bar toughness. Swinden and Bolsover<sup>1</sup> obtained the following results in the case of 0.5 per cent carbon steel oil quenched from 840°C. in the form of 1½" diameter bar and tempered at 650°C. :

Grain size	Tensile strength tons/sq. in.	Yield stress tons/sq. in.	Elongation %	Reduction of area %	Izod impact ft. lb.
Coarse	53.1	37.0	25.5	59.2	16.7
Fine	50.0	35.1	27.0	61.6	76.0

The impact toughness of the fine grained steel was distinctly superior. When this fine grained steel after quenching was tempered at 600°C., it gave the following values :

Tensile strength tons/sq. in.	Yield stress tons/sq. in.	Elongation %	Reduction of area %	Izod impact ft. lb.
53.4	...	26	57.2	54.7

The tensile strength was the same as that of coarse grained steel but the izod value was much higher. Scott<sup>2</sup> has shown that when the coarse and fine grained types of the same steel are heat treated to give the same hardness value, say between 400 and 600 V.P.H.No., the toughness of the fine grained type is much greater than that of the coarse grained.

Grain size	Hardness value	Izod impact value ft. lb.
Fine	440	37
Coarse	440	8
Fine	600	25
Coarse	600	6

Davenport and Bain<sup>3</sup> took one carbon tool steel, heated to different temperatures above the critical temperature range to develop different austenitic grain sizes, quenched from a uniform temperature and tempered at different temperatures to give a Rockwell hardness of C 50. The results obtained are given below :

Heating temperature °C.	Quenching temperature °C.	Austenitic grain size developed at the heating temperature	Hardness C Rockwell	Impact ft. lb.
760	760	Very-fine	50	11
845	760	Fine	50	3
930	760	Coarse	50	2
1010	760	Very coarse	50	1



FIG. 1



FIG. 2



FIG. 3

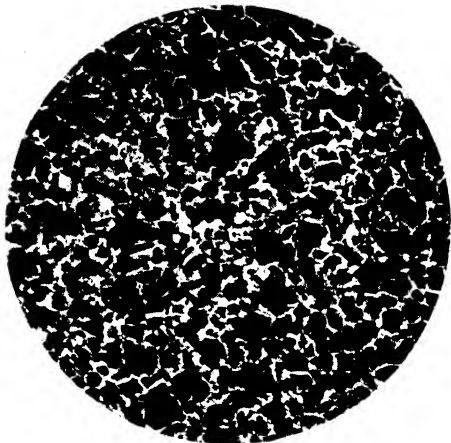


FIG. 4

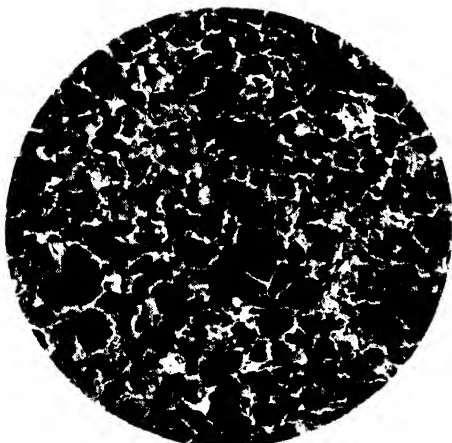


FIG. 5

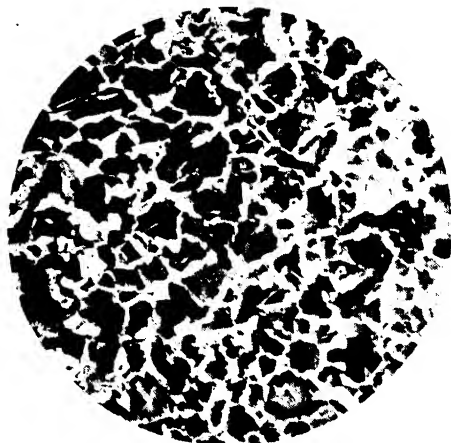


FIG. 6

FIGS. 1-6 — NITAL ETCH. (  $\times 100$  ).

TABLE I

No.	MATERIALS	CHEMICAL COMPOSITION							TENSILE PROPERTIES				
		Carbon %	Manga- nese %	Silicon %	Sulphur %	Phos- phorus %	Nickel %	Chro- mium %	Maximum stress tons/sq. in.	Yield stress tons/sq. in.	% elon- gation	% reduction of area	
Set I:													
1	Tyre steel, mileage run 36,342 miles by tyre	0.56	0.53	0.405	0.035	0.027	nil	0.57	57.88	26.31	14.06	30.72	
5	Tyre steel, mileage run 295,570 miles by tyre	0.60	0.57	0.34	0.036	0.035	nil	nil	60.42	28.32	14.06	31.80	
10	Tyre steel, mileage run 312,656 miles by tyre	0.59	0.58	...	0.040	0.036	nil	0.57	58.61	28.02	14.84	32.03	
11	Tyre steel, mileage run over 350,000 miles by tyre	0.63	0.88	...	0.039	0.025	nil	nil	57.4	25.02	13.28	25.14	
12	Tyre steel, mileage run 445,668 miles by tyre	0.62	0.72	0.348	0.038	0.038	nil	nil	53.63	25.11	16.4	26.71	
Set II													
A	Tyre steel	0.58	0.62	0.385	0.043	0.030	nil	nil	50.60	23.08	18.75	30.16	
B	Tyre steel	0.56	0.62/3	0.415	0.042	0.023	nil	nil	49.17	26.49	17.96	34.04	
C	Wheel steel	0.49/50	0.70	0.360	0.038	0.051	nil	nil	46.86	21.87	17.18	35.83	
D	Wheel steel	0.50	0.66/5	0.347	0.045	0.048	nil	nil	45.95	23.10	21.09	39.50	
E	Wheel steel	0.51	0.68	0.350	0.048	0.045	nil	nil	51.28	23.27	15.62	28.62	

From a correlation of the grain structures revealed by the McQuaid-Ehn carburizing austenitic grain tests as also to some extent by the normalized structures, it would be observed that with identical steel compositions, the coarse McQuaid-Ehn grain size is associated with much lower izod impact value, slightly higher tensile and yield strengths, lower percentage elongation or reduction of area and slightly higher hardness value than the fine McQuaid-Ehn grain size steel. This contrast in the properties is appreciably shown by steel No. E (Set II) possessing coarse grain size in relation to steel Nos. C and D (Set II) showing fine grain sizes. Steel No. E (Set II), although of practically the same composition as C and D (Set II), possesses a lower izod impact toughness, slightly higher tensile strength, lower percentage elongation and reduction of area, and slightly higher hardness than the fine grained steel Nos. C and D (Set II).

The same contrast in physical properties and austenitic grain sizes is afforded by fine grained steel Nos. A and B (Set II) and coarse grained Nos. 5, 10 and 1 (Set I). These two different makes of steel are of identical composition except for slight residual chromium in case of steel Nos. 1 and 10. Steel No. 5 (Set I) was of practically the same composition as steel Nos. A and B (Set II). Set I steels possessed coarse austenitic grain sizes and gave somewhat higher tensile and yield strengths, lower percentage of elongation and reduction of area, higher

TABLE I

PHYSICAL TESTS						METALLURGICAL EXAMINATIONS					
IZOD IMPACT VALUES				BEND TEST		HARDNESS TEST		Micro examination of normalized structures	Inclusion count determination (average of 5 fields, on longitudinal sections at (x100), field diameter 8 cm.)		Mcquaid-Ehn austenitic carburizing grain-size structures
1st notch ft. lb.	2nd notch ft. lb.	3rd notch ft. lb.	Average ft. lb.	Longitudinal, angle of bend on fracture	Transverse, angle of bend on fracture	Longitudinal, V.P.H. No.	Transverse, V.P.H. No.		Elon-gated type	Globu-lar type	
4	4	4	4	...	33°	266, 265, 267, average 266	263	Sorbo-pearlitic grains with ferrite mesh work. Normalized structure (Fig. 1)	4-6	1-3	Fairly coarse grain structure (Fig. 9, sodium picrate etching)
3.5	4	4	3.83	62°30	39°	263, 263, 263, average 263	262	do	6-10	1-2	Coarse grain size (Fig. 10, sodium picrate etching)
5	5	5.5	5.16	54°30	31°	266, 268, 262, average 265	260	do	6-10	1-2	Coarse grain structure (Fig. 11, sodium picrate etching)
5	5	5.5	5.16	36°	20°	262, 269, 269, average 266	262	Coarse sorbo-pearlitic grain structure with ferrite network (Fig. 2)	6-10	1-2	Coarse grain structure (Fig. 12, sodium picrate etching)
0.5	3	5	4.83	40°	20°	236, 232, 236, average 234	236	Coarse sorbo-pearlitic grain structure with ferrite network (Fig. 3)	4-6	1-3	Coarse grain structure (Fig. 13, sodium picrate etching)
10.5	11	10	10.5	61°30	52°	216, 215, 216, average 216	225	Very fine sorbo-pearlitic grain structure with boundary ferrite enclosures (Fig. 4)	4-6	1-3	Fine grain size in general with area of duplexed grain structure. (Former grain structure, Figs. 14 & 15; latter, Figs. 16 & 17, sodium picrate etching)
12	10.5	10	10.83	104°	24°	216, 216, 216, average 216	217	Very fine sorbo-pearlitic grain enclosed in boundary ferrite (Fig. 5)	4-8	4-6	do
10	10	10	10	94°	49°	208, 209, 207, average 208	210	Very fine sorbo-pearlitic grains with boundary ferrite (Fig. 6)	2	6-10	do
15	14	14	14.3	93°	45°	204, 205, 204, average 204	208	Very fine sorbo-pearlitic grain structure with ferrite enclosures (Fig. 7)	4-6	1-2	do
6	6	6.5	6.16	50°	31°	235, 235, 235, average 235	235	Extremely coarse sorbo-pearlitic grain structure with ferrite boundaries (Fig. 8)	6	1-2	Coarse grain structure in general (Fig. 18 sodium picrate etching)

hardness values and fairly lower izod impact values in relation to the finer austenitic grain-size steels Nos. A and B of Set II which gave somewhat lower tensile and yield strengths and hardness values, higher percentage elongation and reduction of area and fairly higher izod impact values.

The transverse bend tests give a good indication of the relative inclusion contents of the various steels — the longitudinal bend test, however, is a function of the relative ductility of the materials. The excellent physical set of properties of the steels of Set I after 300,000 - 400,000 of service mileage should dispel the doubts of engineers who fear that steel gets "fatigued" or "deteriorates" during prolonged service in quality.



FIG. 7

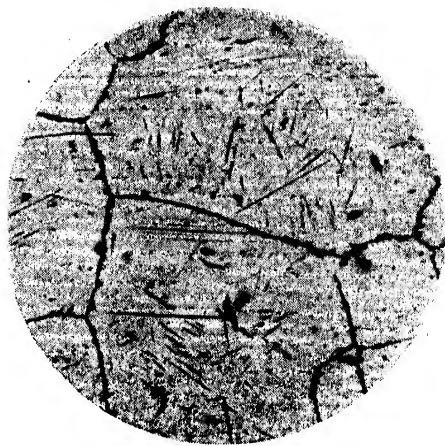


FIG. 8

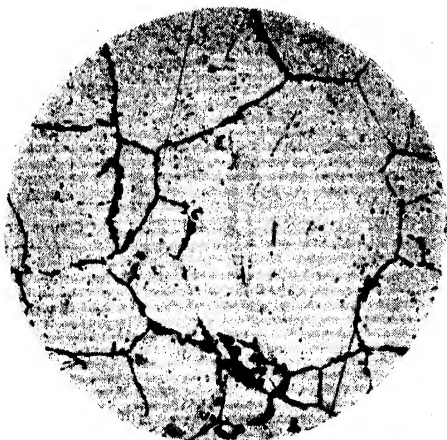


FIG. 9



FIG. 10



FIG. 11

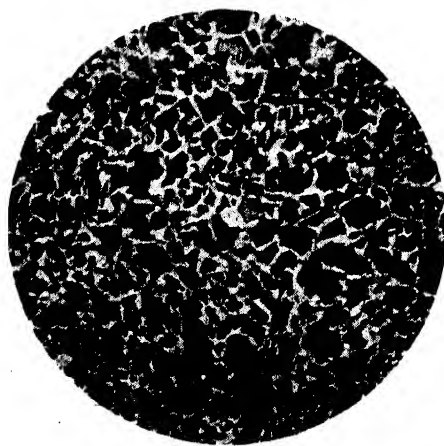


FIG. 12

FIGS. 7-8 — NITAL ETCH. ( $\times 100$ ). FIGS. 9-12 — SODIUM PICRATE ETCH. ( $\times 100$ ).

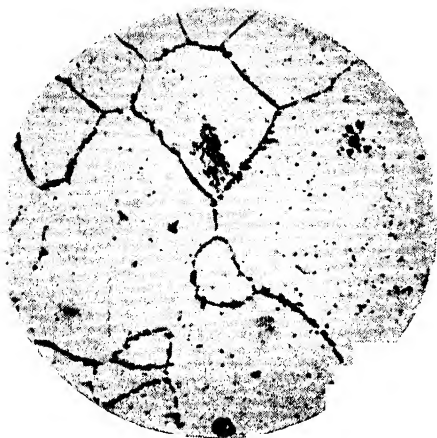


FIG. 13

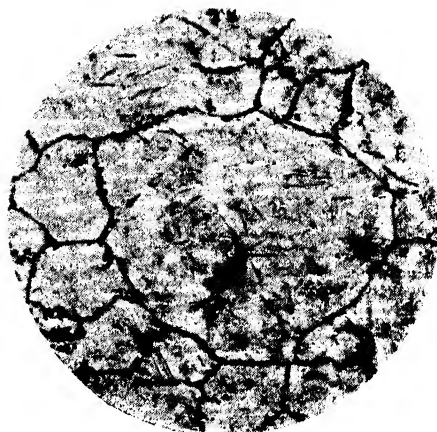


FIG. 14

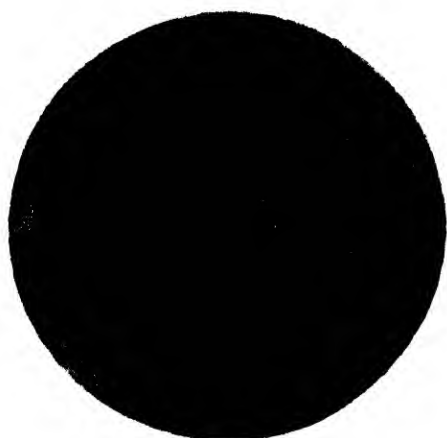


FIG. 15

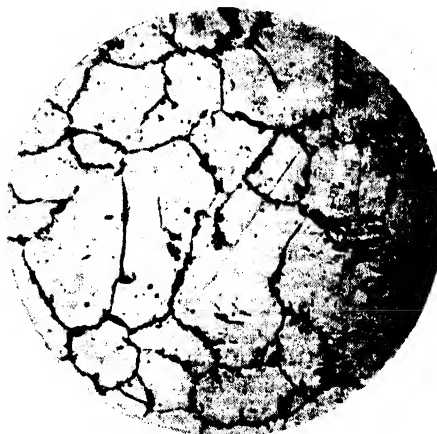


FIG. 16



FIG. 17



FIG. 18

FIGS. 13-18 — SODIUM PICRATE ETCH. (  $\times 100$  ).



These results show that the austenitic grain size established during heating greatly influences the toughness of the heat-treated steel.

O'Neill<sup>4</sup> gave the following results for straight carbon steels in coarse and fine grain condition :

	A %	B %
Carbon	0.35	0.35
Silicon	0.22	0.25
Manganese	0.95	0.96
Sulphur	0.027	0.027
Phosphorus	0.036	0.036
Nickel	0.44	0.46
Chromium	0.06	0.09
Aluminium	0.001	0.009
Grain	Coarse	Fine
Yield stress, tons/sq. in.	29.4	30.4
Max. stress, tons/sq. in.	44.6	43.4
Elongation, %	64	66
Izod value, ft. lb.	12	93

### Properties of Railway Materials

The materials examined consisted of 2 sets. Set No. I comprising of railway locomotive tyres fabricated about a couple of decades ago and which had given excellent service mileage of, in some cases, 300,000 to 400,000 miles under heavy traffic loads. Set No. II was made up of railway tyres ready to go into service. The steels of Set No. I contained but traces of aluminium in solution of the order of 0.002 per cent while those of Set No. II contained between 0.008-0.01 per cent of dissolved aluminium except steel No. E, which showed an aluminium content of the same order as that of Set No. I.

Table I gives the results of the chemical, physical and metallurgical tests carried out. For each test, test pieces of standard dimensions were taken. To start with, a block of standard size ( $10" \times 1\frac{1}{2}" \times 4"$ ) was cut out of the railway tyres and wheels. The blocks were then "normalized" under standard conditions. From such blocks standard dimensioned test pieces were machined out for each of the physical and metallurgical tests.

**Hardenability** — The properties of quenched steels are more directly related to their austenitic grain size than those of annealed or normalized steels. It is necessary to differentiate between the potential maximum hardness attainable and hardenability or the depth of hardening of the mass of the metal. The maximum surface hardness attained on quenching does not greatly depend upon the austenitic grain size for a given mass but the depth of hardening is influenced by the austenitic grain size to a marked degree. This is illustrated in Fig. 19.

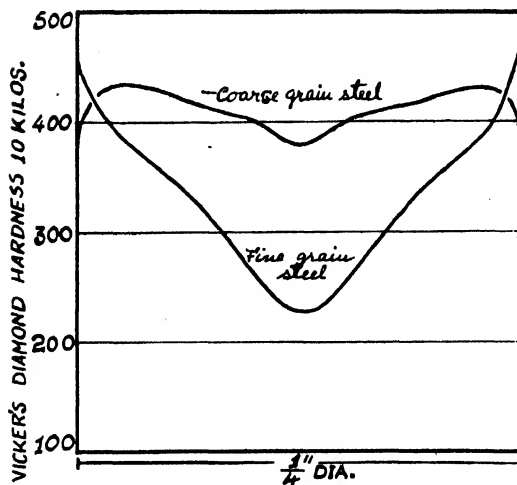


FIG 19 — VARIATIONS IN HARDENABILITY WITH GRAIN SIZE : FINE GRAINED STEEL CONTAINED 0.02 PER CENT ALUMINIUM WHILE THE COARSE GRAINED WAS ALUMINIUM FREE. BOTH STEELS WERE QUENCHED FROM 980°C.

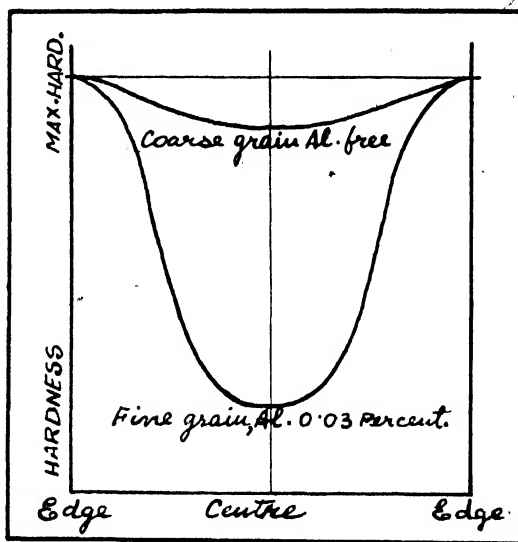


FIG. 20 — VARIATION OF HARDENABILITY WITH GRAIN SIZE : UPPER CURVE REPRESENTS A DEEP HARDENING COARSE GRAINED STEEL AND THE LOWER CURVE A FINE GRAINED SHALLOW HARDENING STEEL IN WHICH THE HARDNESS DROPS TO A VERY LOW VALUE IN THE CORE. THE SURFACE HARDNESS IS THE SAME IN BOTH THE CASES. BOTH THE STEELS WERE QUENCHED FROM THE SAME TEMPERATURE.

In general, the fine grained steels are shallow hardening and the coarse grained types are deep hardening and less subject to mass effect (FIG. 20).

Due to large austenitic grain boundary surface areas in the case of fine grained steels, the transformation rates of these steels on quenching are faster than the coarse grained types. So in the case of the former the core undergoes comparatively rapid transformation and liberates more of the ferrite which makes it less hard and renders the steel shallow hardening.

Since the toughness or impact values of hardened steels increase as the hardness diminishes, fine austenitic grained steels are not so hard in the core as coarse austenitic grained steels. Therefore, they have tougher cores and resist dynamic stresses to a greater extent than similar deep hardening coarse austenitic grained steels. Fine grained plain carbon steels can be economically substituted for low alloy steels where high core toughness is important. However, in tool and certain other types of steels where deep hardening properties are essential, the fine grained steel is unsuitable. Alloying elements like manganese, etc., add to the deep-hardening properties of the steel.

Fine grained steels are less liable to macroscopic cracking during quenching. Cases are on record where, by making the steel inherently fine grained through suitable deoxidation, the rejections due to cracking in the heat treatment were entirely eliminated. Fine grained steels are also less liable to warping and distortion during heat treatment. It has been further established that the micro-cracks observed in quenched steels result from large dimensional changes that increase with increasing grain size. Thus, the superior toughness of fine grained steels is due, to some extent at least, to the existence of a smaller number of microscopic cracks. Coarser grained steels have a higher internal stress after quenching and are, therefore, more susceptible to grinding cracks than the finer grained types.

However, due to their more rapid rates of transformation, finer grained steels are more prone to soft spots on quenching and are not suitable where full uniform hardening throughout the section is desired.

*Machinability & Surface Finish & Forgeability & Pressing*—The view is widely held in America that coarse grained steels machine better than fine grained. It is stated that in the coarse grained steels the size of the final ferrite and carbide lamellae resulting from the transformation of austenite is coarser and hence better machin-

ability is imparted. However, completely identical results have not been obtained. Where there is a great difference in the machinability as in the case of free cutting and ordinary steels, it is easily perceptible, but when the difference is small, it is difficult to discover which of the two types possess superior machining properties. However, the coarse grained structure causes roughness of the finished surface and the fine grained steels give a superior finish. In punch press operations fine grained steels exhibit several advantages over the coarse grained types. It has been shown that the finished surfaces of fine grained mild steels were much superior to those of coarse grained steels since the latter became rough after deep drawing whereas the fine grained steel remained smooth and uniform. In blanking, fine grained types shear cleaner and exhibit smaller burrs and are less embrittled by cold work and, therefore, may be drawn deeper and show fewer defects than the coarse grained steels. Fine grained steels are more suitable for heavy punching operations, drastic cold rolling, shearing and trimming.

Coarse grained steels forge more readily than fine grained types due to low interference to slip in the coarse grained types and coarse grained types give better fibre structures than the fine grained steels on forging.

*Carburizing Characteristics*—It has been observed that the rate of diffusion of carbon into fine grained austenite is less rapid than in the coarse grained austenite and so fine grained types carburize less deeply and more slowly than the coarse grained steels. Fine grained steels are prone to give abnormal or soft spots on quenching after carburization. Abnormal spots refer to a coalesced state of carbide which causes low hardness values. The object of carburization is to obtain components with a case possessing a high surface hardness and of sufficient depth and a soft but tough core. The carbon content of a low carbon steel is usually raised by heating for a sufficient length of time in a carburizing atmosphere, i.e. usually in a container packed with a solid carburizing compound. The carburized material is then quenched to harden the case. Apart from the shallow-hardening characteristics of fine grained steels, these are ideal for carburizing. During the prolonged heating at the carburizing temperature, pronounced grain growth occurs in

steels which have not been suitably de-oxidized and are, therefore, coarse grained. So after case-carburization, grain-refining treatments have to be carried out. The following is a complete cycle :

(a) Carburization and air cooling of the box after carburization.

(b) Heating the material to just above the critical temperature range of the core, about 850°-880°C. to refine its grain size, followed by quenching in oil or water.

(c) Heating just above the critical temperature range of the high carbon case, about 750°-770°C. to refine its grain size, followed by quenching in oil or water.

(d) Final tempering at about 200°C. to relieve the internal stresses.

Where fine grained steels are employed, the core and case retain fine grained characteristics after prolonged heating at the carburizing temperature and hence the core and case grain-refining operations, etc., can be safely omitted and the case-carburized material quenched directly from the carburizing box. In some cases even the final tempering may also be dispensed with. It is now realized that so long as an allowance is made for the slower penetration of carbon in fine grained steels, these have pronounced advantages over the coarse grained types in materially cutting down heat-treatment costs and time, yielding more production of an equally good, if not better, case-hardened material. There is always the danger of warpage, distortion, macro-cracks in the heat-treatment cycle following carburization of the coarse grained steels, e.g. in case-hardened gear applications. Such dangers practically do not exist in fine grained steels.

**"Pipe" : Shrinkage Cavity**—Fine grained steels tend to give a deeper pipe than normal coarse grained steels.

**Decarburization**—Coarse grained steels show a greater tendency towards decarburization than the fine grained types.

**Ageing**—Fine grained steels age much less than coarse grained steels. The coarse grained steels show after ageing an increase in the hardness, tensile strength, yield point and a greater loss in the impact toughness, reduction of area and elongation values than the fine grained steels. In one case,

the impact toughness of a fine grained steel was actually shown to increase after ageing.

**Temper-brittleness**—Temper-brittleness is the name applied to the loss of ductility or impact toughness that results after tempering hardened specimens to a temperature below the critical range followed by slow cooling. Steels containing alloying elements like nickel, chromium are chiefly susceptible to temper-brittleness. It has been shown by various workers that after tempering in the temper-brittleness range the izod impact values of the fine grained steels are much greater than coarse grained steels although the latter is somewhat less susceptible to temper-brittleness than the fine grain aggregates.

**Magnetic Properties**—Rüder<sup>5</sup> has correlated the existing data of the effect of grain size upon the magnetic properties of steels and has shown that hysteresis, coercive force and residual magnetism all decrease with increase in grain size. This condition varies with prior treatments, whether mechanical or chemical, and consequently the effect of grain size cannot readily be ascertained.

#### Grain Size Control & Its Practical Applications

A full understanding of the characteristics of fine and coarse grained steels has led to the choice of each type for different purposes. Coarse grained steels are recommended for easy machinability, deep hardening, etc., and fine grained steels are preferred for advantages hitherto obtained from alloying elements, e.g. greater toughness with given strength and hardness, wide heat-treating range, freedom from danger of cracking, warpage, etc., and the elimination of grain-refining treatments after carburizing. All these attributes should be taken into consideration for the purpose of selection of the steel for a particular purpose.

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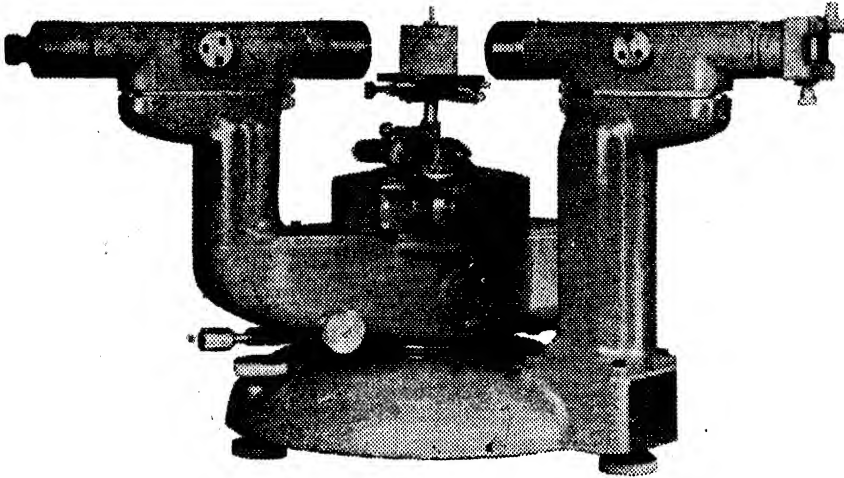
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# Chemical Examination of the Oil from the Seeds of *Gmelina asiatica* Linn.

J. S. AGGARWAL & (Miss) PADMINI SONI

Chemical Laboratories, Council of Scientific & Industrial Research, Delhi

**G**MELINA ASIATICA Linn., commonly known as *Badhara* in Hindi, belongs to the natural order Verbenaceae.

It is a large and much-branched shrub of the forests of South India, Burma and Ceylon. It is also cultivated in Bengal. The root has been used as a demulcent and alterative<sup>1</sup>, and in the treatment of rheumatic pains and syphilitic diseases<sup>2</sup>. But for a paper on the chemistry of the oil from the seeds of *Tectona Grandis* (Teak) by Puntambekar and Krishna<sup>3</sup>, no work on the seed oils from the plants of this natural order has been reported in the literature. Puntambekar and Krishna have not reported results on the separation and identification of individual acids by fractional crystallization or by fractional distillation of their methyl esters. It was, therefore, considered of interest to undertake the chemical examination of the fatty oil from the seeds of *Gmelina asiatica* Linn., which belongs to the same natural order.

The seeds of *Gmelina asiatica* Linn. contain about 7.5 per cent of fatty oil with the following fatty acid composition: palmitic acid, 9.57 per cent; stearic acid, 19.67 per cent; linoleic acid, 25.83 per cent; oleic acid, 33.64 per cent; and ricinoleic acid, 11.29 per cent. The unsaponifiable fraction of the oil consists of sitosterol and a small amount of yellowish-orange colouring matter.

The oil belongs to the semi-drying class.

## Experimental

Powdered *badhara* seeds were extracted 4 times with petroleum ether (40°-60°C.).

After removing the solvent about 7.5 per cent of a greenish-yellow oil having the following characteristics was obtained:

Specific gravity at 40°C.	0.9132
Viscosity at 40°C.	34.1 centistokes
Refractive index at 40°C.	1.4645
Acid value	10.45
Saponification value	191.75
Iodine value (Wijs)	100.4
Acetyl value	11.8
Hehner value	93.12
Hexabromide value	nil
Unsaponifiable matter	2.20%
Soluble fatty acids calculated as butyric acid	0.94%
Saturated fatty acids (modified Bertram method) <sup>4</sup>	28.2%

250 gm. of the oil were saponified with an alcoholic solution of caustic potash and the soap dissolved in water. The unsaponifiable matter was removed with ether and the soap solution decomposed with dilute sulphuric acid. When heated on the water bath, the fatty acids formed a clear oily layer at the top. They were removed by extraction with ether and dried in vacuum at 100°C.

The acids (200 gm.) were dissolved in 95 per cent alcohol (1,000 c.c.), the solution was boiled and mixed with a boiling solution of lead acetate (140 gm.) in 95 per cent alcohol (1,000 c.c.) containing 1.5 per cent glacial acetic acid. The insoluble lead salts obtained on cooling at 15°C. overnight were crystallized from alcohol containing 1.5 per cent glacial acetic acid and recrystallized from ether at 0°C. when an almost white crystalline material was obtained. The solid acids were regenerated from these lead

salts. The liquid acids were recovered from the lead salts left on the evaporation of the mixed ether and alcohol mother liquors. The amounts of soluble and insoluble lead salt fatty acids as well as some of their characteristics are given in Table I.

TABLE I

FATTY ACIDS	AMOUNT %	NEUTRAL- IZATION VALUE	IOD. VALUE	ACETYL VALUE
Total	...	197.7	101.4	15.0
Soluble lead salt	71.9	193.0	116.5	25.4
Insoluble lead salt	28.1	205.9	1.1	nil

#### Soluble Lead Salt Fatty Acids

The soluble lead salt fatty acids on oxidation with potassium permanganate (modified Bertram method<sup>4</sup>), gave 1.5 per cent saturated acid, identified as palmitic acid.

The liquid acids were converted into methyl esters, and a known weight was fractionally distilled under reduced pressure<sup>5</sup>. The temperatures and pressures maintained during the distillation and the weights of the fractions obtained are given in Table II.

TABLE II

Weight of ester distilled, 57.0 gm.

FRACTION No.	TEMPERATURE °C.	PRESSURE mm.	WEIGHT OF FRACTION gm.
L-1	185-190	9.0	7.51
L-2	190-198	9.0	6.89
L-3	198-205	9.0	7.97
L-4	205-207	9.0	8.52
L-5	207-210	9.0	7.49
L-6	212-220	10.0	7.85
L-7	220-230	10.0	4.14
L-8	230-240	10.0	3.80
L-9	Residue	...	1.93
			55.10

The loss in distillation (1.9 gm.) was proportionally divided and added to each fraction. The saponification and iodine values were determined and the amounts of individual esters in each fraction were

calculated. The results obtained are given in Table III.

The acids from L-3 and L-6 were oxidized with dilute alkaline potassium permanganate<sup>6</sup> when tetrahydroxy stearic acid (m.p. 173°C.) and dihydroxy stearic acid (m.p. 130°C.) were isolated from both the fractions. Acids from L-7 gave trihydroxy stearic acid (m.p. 110°C.) by this treatment. The composition of soluble lead salt fatty acids is as follows:

	%
Palmitic acid	1.67
Linoleic acid	36.04
Oleic acid	46.27
Ricinoleic acid	16.02

The bromine addition derivatives of the soluble lead salt fatty acids were prepared according to the method of Eibner and Muggenthalor<sup>7</sup> as described by Jamieson and Boughmann<sup>8</sup>. The results are as follows:

Soluble lead salt acids taken for analysis	3.3157 gm.
Palmitic acid from Bertram's method	0.05 gm.
Ricinoleic acid calculated from acetyl value	0.5106 gm.
Linoleic tetrabromide insoluble in petrol ether, m.p. 114°-115°C.	1.2850 gm.
Residue ( tetrabromide, dibromide of oleic and ricinoleic acids and palmitic acid )	4.5688 gm.
Bromine content of the residue	40.29%
Linoleic tetrabromide in the residue	1.2589 gm.
Total tetrabromide found	2.5439 gm.
Linoleic acid equivalent of tetrabromide, 1.1872 gm.	35.8%
Oleic acid in the liquid acids	47.3%

The mean values obtained for these acids from distillation and bromination results are:

	%
Palmitic acid	1.58
Linoleic acid	35.92
Oleic acid	46.79
Ricinoleic acid	15.71

TABLE III

FRACTION No.	CORRECTED WT.	IOD. VALUE	SAP. VALUE	MEAN MOL. WT.	WEIGHT OF ESTERS			
					Methyl palmitate gm.	Methyl linoleate gm.	Methyl oleate gm.	Methyl ricinoleate gm.
L-1	7.76	98.14	194.52	288.4	0.9	2.0	4.86	...
L-2	6.61	110.07	190.2	294.9	0.06	1.91	4.64	...
L-3	8.24	127.2	190.04	295.22	...	3.94	4.30	...
L-4	8.81	137.2	190.17	295.0	...	5.23	3.58	...
L-5	7.75	145.8	190.29	294.8	...	5.37	2.38	...
L-6	7.64	109.5	189.84	295.5	...	2.10	5.54	...
L-7	4.29	81.5	181.08	309.8	...	...	0.54	3.75
L-8	3.90	81.5	180.97	310.0	...	...	0.50	3.40
L-9	2.00	81.0	179.81	312.0	...	...	0.04	1.96
Total	57.00	...	...	...	0.96	20.55	26.38	9.11

**Insoluble Lead Salt Fatty Acids**

The insoluble lead-salt acids after conversion into methyl esters were distilled under reduced pressure. The fractions obtained are given in Table IV.

**TABLE IV**  
Weight of esters distilled, 46.5 gm.

FRACTION	TEMPERATURE RANGE °C.	PRESSURE mm.	WEIGHT OF FRACTION gm.
S-1	185-190	3.4	9.4
S-2	192-196	3.4	13.23
S-3	196-200	3.4	10.88
S-4	200-202	3.4	6.4
S-5	202-205	5	3.99
S-6	Residue	...	1.30
			45.20

From the above analysis the total fatty acid constituents of the seeds of *Gmelina asiatica* Linn. are as follows :

Palmitic acid	9.57
Stearic acid	19.67
Linoleic acid	25.83
Oleic acid	33.64
Ricinoleic acid	11.29

The composition of the oil, therefore, is the following :

	%
Palmitic acid	9.41
Stearic acid	19.27
Linoleic acid	25.18
Oleic acid	32.95
Ricinoleic acid	10.99

Unsaponifiable matter consisting of sitosterol and a yellowish-orange colouring matter 2.2

**TABLE V**

FRACTION No.	CORRECTED WT. gm.	IOD. VALUE	SAP. VALUE	MEAN MOL. WT.	WEIGHT OF ESTERS		
					Methyl palmitate gm.	Methyl stearate gm.	Unsaturated esters gm.
S-1	9.67	0.68	196.14	285.9	4.3	5.31	0.06
S-2	13.60	1.02	194.75	287.07	5.03	8.45	0.12
S-3	11.18	1.10	192.55	291.2	2.84	8.23	0.11
S-4	6.59	1.14	191.00	293.71	1.1	5.43	0.06
S-5	4.11	1.20	190.29	294.90	0.53	3.54	0.04
S-6	1.35	1.11	189.00	296.82	0.08	1.26	0.01
Total	46.50	.....	.....	.....	13.88	32.22	0.40

The loss in distillation (1.3 gm.) was added proportionally to each fraction. The iodine values, saponification values and the mean molecular weights of all the fractions were determined and the amounts of the various acids in the different fractions calculated according to the method of Jamieson and Boughmann<sup>8</sup> (Table V).

The acids from fraction S-1 on repeated crystallization from alcohol and acetone gave a product melting from 60° to 66°C. showing it to be a mixture of palmitic acid (m.p. 63°C.) and stearic acid (m.p. 69°C.). The acids from fraction S-5 on repeated crystallization from acetone gave stearic acid.

The amount of individual saturated components in the insoluble lead salt fatty acids, therefore, are as follows :

	%
Palmitic acid	30
Stearic acid	70

**Acknowledgement**

The authors record their grateful thanks to Dr. S. Siddiqui for his interest in this investigation.

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# Metabolism of Carotenoid Pigments in Plant Leaves During Development

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EARLIER work on the carotenoids of leaves showed that carotene begins to develop soon after the germination of the seed<sup>1-3</sup>. After germination the carotene content increases with the growth of the plant until the plant reaches maturity when it begins to decrease<sup>4-7</sup>. Ramanov<sup>8</sup> reported decrease in carotene content of leaves in the early stages of growth. Beck<sup>9</sup> reported that both xanthophyll and carotene increased with the age of the plant even though grown in the dark, but the rate of development decreased with time. Mackinney<sup>10</sup> has shown that there is apparently no significant change in the ratio of  $\alpha$  :  $\beta$ -carotene with the degree of development in the leaves of English ivy and the coastal red wood. Virtanen and co-workers<sup>11</sup> reported that the total quantity of carotene of a plant increased rapidly up to the time of blooming and then diminished as the plant matured. Hauge<sup>12</sup> found that alfalfa, when 10"-12" high, contained almost twice as much  $\beta$ -carotene as when it was in full bloom. This observation has been confirmed by Hilton, Hauge and Wilbur<sup>13</sup> who found in young alfalfa, 10"-12" high, 90 units of  $\beta$ -carotene per gm. while it was only 70 units in the bloom stage. They also noticed that soyabean, 12"-15" high, contained 45 units of  $\beta$ -carotene per gm. while the more mature plant, as cut for hay, contained only 30 units. That grasses showed a steady decline in the carotene values with increasing maturity was shown by Wall<sup>14</sup> and Seshan and Sen<sup>15</sup>. In oats and barley plants, carotene continues to decrease during the period of ripening and ultimately reaches low values<sup>16</sup>. Esselen and co-workers<sup>17</sup> found that the  $\beta$ -carotene content of maize plant increased as the plant reached full growth after which there was a marked loss of  $\beta$ -carotene. Similar observations have been made by Snyder and Moore<sup>18</sup>, who found that the carotene content of alfalfa, brom grass, corn leaves, oat plant and soyabean plant is much greater during the earlier stages of growth than after they

reach the stage of maturity. Bondi and Meyer<sup>19</sup> reported that the leaves of both cultivated and wild plants including those of fodders and vegetables, as a rule, contained higher amount of carotene if grown in winter than when grown in summer, and further the carotene content declined during growth.

The work referred to above deals primarily with the development of  $\beta$ -carotene during the growth of plant tissues. The formation of the various carotenoid pigments and the changes that occur in each one of them at various stages of the development of the plant tissues have not been fully studied. It is possible that the development of all the pigments do not follow the same pattern. The metabolic inter-relationships of different carotenoids which a systematic investigation could reveal are likely to be of value in reaching an understanding of the functions of these pigments in the plants. The leafy tissues of six plants, viz. rape seed, fenugreek, spinach, lettuce, cauliflower and carrots have been investigated.

## Experimental

The plants were grown and maintained under uniform conditions, as far as possible, for the duration of the experiment. The leafy vegetables were grown in small plots, and the conditions of soil, manuring, irrigation and climatic factors were uniform for all the plants. The specimens were drawn from plants of the same age and differences obtained in the quantities of carotenoids in different species are, therefore, to be attributed to the characteristics of the species.

The carotenoids were determined in samples of fresh material soon after collection. Samples were collected twice or thrice weekly from the early stages for about two months. Leaves were collected at random from the plant, wrapped in a black paper and immediately taken to the laboratory for analysis. About 5 gm. of a representative sample were quickly weighed and immediately covered with alcohol. A small

TABLE I—CAULIFLOWER

No.	Date of examination	No. of days from the commencement of the experiment	Total pigments	Xanthophyll		Unidentified pigment of band II		Neo-β-carotene U		β-carotene		Neo-β-carotene B		Total active pigments in terms of β-carotene		Vitamin A potency I.U./gm.
			μgm./gm.	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	
1	Feb. 13	...	189.9	125.2	65.9	10.5	5.5	7.9	4.2	37.5	19.8	8.8	4.6	43.87	23.1	73.1
2	" 19	6	202.5	131.6	65.0	12.5	6.2	8.0	4.0	40.6	20.0	9.8	4.8	47.5	23.0	79.2
3	" 25	12	201.0	121.5	60.4	14.8	7.3	10.1	5.0	44.8	22.3	9.8	4.9	52.22	26.0	87.0
4	" 28	15	264.9	158.8	59.9	16.4	6.2	8.0	3.7	70.6	26.7	11.1	4.2	78.15	29.5	130.3
5	Mar. 3	18	303.7	188.7	62.1	18.0	5.9	14.5	4.8	72.8	24.0	9.7	3.2	81.27	26.8	135.5
6	" 8	21	299.7	209.7	69.7	9.0	3.0	7.9	2.6	67.3	22.5	6.6	2.2	72.57	24.2	121.0
7	" 10	25	290.6	184.9	63.6	13.5	4.6	11.1	3.8	66.4	22.8	5.1	2.2	76.52	25.3	127.5
8	" 13	28	208.2	114.8	61.8	10.5	5.0	10.4	5.1	45.3	21.8	13.4	6.4	54.6	26.2	131.0
9	" 18	33	185.3	114.8	62.0	9.2	5.0	7.0	3.8	41.9	22.6	12.4	6.7	40.85	26.9	83.1
10	" 22	37	181.4	111.9	61.7	12.5	4.3	10.0	5.5	43.5	20.9	13.5	1.9	47.75	26.3	79.6
11	" 23	40	165.3	105.5	63.8	7.1	4.3	6.0	3.6	35.8	20.8	2.4	1.5	47.0	25.4	78.5
12	" 25	43	159.0	100.7	63.3	22.5	14.2	...	...	26.3	15.9	...	...	35.8	22.5	59.7
13	" 31	46	155.9	100.9	64.7	28.7	18.4	...	...	18.1	15.9	...	...	16.3	16.9	43.8
14	Apr. 1	47	113.8	68.3	60.4	27.4	24.1	...	...	15.2	13.8	...	...	15.2	13.8	30.2
15	" 2	49	110.5	66.8	60.4	28.5	25.8	...	...	15.3	13.2	...	...	15.3	13.2	25.3
16	" 7	53	113.6	65.3	56.6	35.0	30.3	...	...	13.0	13.1	...	...	13.0	13.1	23.5
17	" 8	54	99.1	54.7	55.2	31.4	31.7	...	...	9.9	11.9	...	...	9.9	11.9	21.7
18	" 10	56	83.1	45.9	55.2	27.3	32.8	...	...	...	...	...	...	...	...	16.5

TABLE II—FENUGREEK

No.	Date of examination	No. of days from the commencement of the experiment	Total pigments	Xanthophyll		Unidentified pigment of band II		Neo-β-carotene U		β-carotene		Neo-β-carotene B		Total active pigments in terms of β-carotene		Vitamin A potency I.U./gm.
			μgm./gm.	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	μgm./gm.	%	
1	Feb. 10	...	530.4	350.4	66.1	22.1	4.1	19.9	3.7	127.7	24.1	10.3	2.0	137.8	26.0	229.7
2	" 14	6	568.9	338.0	60.5	37.8	6.7	29.1	5.2	132.0	23.6	22.0	3.9	150.3	26.9	250.5
3	" 19	11	564.8	363.6	64.4	27.4	4.9	26.0	4.6	133.2	23.6	14.6	2.6	147.0	26.0	245.0
4	" 23	15	607.5	367.3	60.5	35.0	5.8	26.2	4.3	156.6	25.8	12.4	3.7	169.3	27.8	282.2
5	Mar. 3	21	624.9	367.9	58.7	28.0	6.1	34.0	5.4	170.0	27.2	16.0	2.6	186.5	29.1	310.8
6	" 6	24	652.2	397.2	60.9	38.2	5.8	29.1	4.4	177.3	27.2	10.4	1.8	189.8	29.1	316.3
7	" 8	26	653.1	384.5	58.9	37.1	5.7	34.5	4.5	178.4	27.3	24.0	3.7	197.7	30.3	329.5
8	" 10	28	653.1	345.0	59.3	31.0	5.3	20.1	3.5	160.4	27.7	23.8	4.1	177.1	30.6	295.2
9	" 14	32	579.9	382.0	60.5	52.0	8.2	20.1	4.7	153.0	24.1	17.0	2.7	169.0	28.6	281.7
10	" 20	38	590.7	362.8	61.4	35.0	5.9	20.1	3.4	158.6	26.8	14.2	2.4	170.7	28.9	294.5
11	" 24	42	553.3	332.2	60.0	32.0	5.8	27.0	4.8	136.1	24.8	26.0	4.7	155.8	28.1	259.7
12	" 29	47	534.4	292.3	54.7	49.7	9.3	34.0	7.1	135.2	25.3	19.2	8.6	154.3	28.9	257.2
13	Apr. 4	53	456.6	247.5	54.2	29.1	6.3	17.2	3.8	133.0	29.1	29.8	6.5	152.2	33.3	253.7

TABLE III—CARROT

No.	DATE OF EXAMINATION	No. OF DAYS FROM THE COMMENCEMENT OF THE EXPERIMENT	TOTAL PIGMENTS	XANTHOPHYLL		UNIDENTIFIED PIGMENT OF BAND II		NEO- $\beta$ -CAROTENE U		$\beta$ -CAROTENE		NEO- $\beta$ -CAROTENE B		TOTAL ACTIVE PIGMENTS IN TERMS OF $\beta$ -CAROTENE		VITAMIN A POTENCY
				$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	
1	Feb. 13	...	395.9	253.1	65.2	23.0	4.0	10.9	2.8	90.9	23.0	13.0	3.3	100.1	25.3	166.8
2	" 19	6	429.9	285.8	66.5	15.9	3.7	10.7	2.5	77.9	18.1	39.6	9.2	100.4	23.3	167.3
3	" 24	11	415.3	270.5	65.1	23.1	5.6	14.7	3.5	89.8	21.6	17.2	4.1	102.1	24.6	170.1
4	" 27	14	493.5	296.3	60.0	54.4	11.0	19.0	3.9	96.4	19.5	27.4	5.6	114.9	23.3	191.5
5	Mar. 3	18	473.2	300.2	63.4	27.2	5.8	13.8	2.9	105.0	22.2	27.0	5.7	121.9	23.7	203.2
6	" 5	20	543.1	353.2	65.0	46.2	8.5	13.0	2.4	97.7	18.0	33.0	6.1	117.4	21.6	195.7
7	" 8	23	546.8	370.1	67.7	27.9	5.1	20.1	3.6	101.9	18.6	26.8	5.0	120.3	22.0	200.5
8	" 12	27	548.4	353.3	64.4	45.6	8.3	18.0	3.3	102.0	18.6	29.5	5.3	121.2	22.1	202.2
9	" 15	30	553.5	346.8	62.7	65.5	11.8	14.5	2.6	97.7	17.7	29.0	5.2	115.8	20.9	193.0
10	" 18	33	413.9	270.4	65.3	18.0	4.3	9.0	2.2	96.2	23.3	20.3	4.9	103.6	26.2	181.0
11	" 21	36	409.5	270.3	66.0	30.2	7.4	11.1	2.7	84.4	20.6	13.5	3.3	94.0	23.0	156.7
12	" 26	41	368.5	252.6	68.5	14.2	3.8	12.4	3.4	77.0	20.9	12.3	3.3	86.2	23.4	143.7
13	" 29	44	436.1	272.8	62.6	45.1	10.3	26.2	6.0	68.6	15.7	23.4	5.4	86.8	19.9	144.7
14	Apr. 3	49	343.8	236.3	68.7	9.0	2.6	16.3	2.0	64.8	18.9	26.9	7.8	79.9	23.2	133.2

TABLE IV—RAPE SEED

No.	Date of examination	No. of days from the commencement of the experiment	Total pigments  $\mu\text{gm./gm.}$	Xanthophyll		Unidentified pigment of band II		Neo- $\beta$ -carotene U		$\beta$ -carotene		Neo- $\beta$ -carotene B		Total active pigments in terms of $\beta$ -carotene  $\mu\text{gm./gm.}$	Vitamin A potency  I.U./gm.	
				$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%			$\mu\text{gm./gm.}$
1	Feb. 14	...	288.6	182.4	63.2	19.0	6.6	15.0	5.2	65.2	22.6	7.0	2.4	72.45	25.1	120.8
2	" 17	3	268.0	172.2	64.3	14.6	5.4	10.7	4.0	64.7	24.1	5.8	2.2	70.28	26.2	117.1
3	" 20	6	292.5	178.2	60.9	15.0	5.1	12.5	4.3	68.1	23.3	18.7	6.4	80.57	27.5	134.3
4	" 24	10	341.9	228.4	66.8	8.0	2.3	8.3	2.4	88.6	25.9	8.6	2.5	94.98	27.8	158.3
5	" 27	13	425.4	258.5	60.8	28.3	6.7	20.2	4.7	92.3	21.7	26.1	6.1	110.4	25.9	184.0
6	Mar. 3	17	407.3	248.5	61.0	20.4	5.0	18.0	4.4	103.4	25.4	17.0	4.2	116.4	28.6	194.0
7	" 4	18	410.3	253.1	61.7	10.3	2.5	10.1	2.4	127.2	31.2	9.6	2.3	134.53	32.6	224.2
8	" 10	24	380.0	235.0	61.8	24.0	6.3	20.8	5.5	88.6	23.3	11.6	3.1	99.6	26.2	166.0
9	" 13	27	334.5	209.8	62.7	11.3	3.4	10.3	3.1	70.1	20.9	33.0	9.9	89.18	26.6	148.6
10	" 17	31	339.8	197.8	58.2	30.7	9.0	20.1	5.9	80.0	23.5	11.2	3.3	90.62	26.7	151.3
11	" 24	38	320.5	192.5	60.0	19.9	6.2	20.0	6.3	71.5	22.3	16.6	5.2	84.8	26.5	141.3

TABLE V — SPINACH

No.	DATE OF EXAMINATION	No. OF DAYS FROM THE COMMENCEMENT OF THE EXPERIMENT	TOTAL PIGMENTS	XANTHOPHYLL		UNIDENTIFIED PIGMENT OF BAND II		NEO- $\beta$ -CAROTENE U		$\beta$ -CAROTENE		NEO- $\beta$ -CAROTENE B		TOTAL ACTIVE PIGMENTS IN TERMS OF $\beta$ -CAROTENE		VITAMIN A POTENCY I.U./gm.
			$\mu\text{gm./gm.}$	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	
1	Feb.	15	151.1	100.3	66.4	11.7	7.7	8.2	5.4	28.5	19.9	2.4	1.6	31.75	21.0	52.9
2	"	19	144.5	96.0	66.4	12.3	8.5	5.4	3.7	27.4	19.0	3.4	2.4	30.45	21.1	50.8
3	"	25	147.4	90.2	61.2	13.1	8.8	10.1	6.8	28.9	19.6	5.1	3.4	33.97	23.0	56.6
4	Mar.	4	173.3	114.2	65.9	12.0	6.9	10.1	4.7	34.5	19.9	4.5	2.6	33.77	22.4	65.6
5	"	8	178.5	109.3	61.2	16.1	9.0	9.3	5.2	39.5	22.1	4.3	2.4	43.97	24.7	73.3
6	"	21	200.6	114.5	57.1	21.2	10.6	13.1	6.5	47.2	23.5	4.6	2.3	53.77	26.8	89.6
7	"	25	213.1	116.7	54.8	20.0	9.4	15.0	5.0	54.4	25.5	7.0	3.3	61.65	28.9	102.8
8	"	10	215.7	120.5	55.9	23.3	10.8	18.2	4.4	48.0	22.3	5.7	2.6	55.4	25.7	92.3
9	"	12	205.0	130.0	63.5	10.1	4.9	7.1	3.5	49.7	24.2	7.9	3.9	55.42	27.0	92.4
10	"	14	178.5	96.0	53.8	14.9	8.3	8.6	4.8	53.0	19.7	6.0	3.4	58.15	32.6	96.9
11	"	17	187.3	101.7	54.3	20.0	10.7	13.9	7.4	45.6	24.3	3.3	3.3	52.12	27.8	86.9
12	"	32	187.3	106.5	58.2	14.5	7.9	8.1	4.4	49.9	27.3	3.9	2.1	53.83	29.4	89.7
13	"	34	182.9	96.9	55.8	20.4	11.7	14.0	8.2	37.3	21.5	5.2	3.0	43.4	25.0	72.3
14	"	22	173.8	79.6	63.6	10.1	8.1	9.2	7.3	22.4	17.9	3.8	3.6	26.6	21.2	44.3
15	"	43	125.1	77.4	60.3	16.1	12.5	10.0	5.9	24.9	19.4	2.7	4	27.4	21.3	45.8
16	"	29	128.4	62.7	61.3	11.9	11.6	6.0	5.9	19.5	19.1	2.2	2.1	22.1	14.2	36.8
17	Apr.	47	102.3	45.1	64.2	15.1	21.5	...	...	10.0	14.2	...	...	10.0	14.2	18.7
18	"	3	70.2	29.9	54.5	18.4	33.6	...	...	6.5	11.9	...	...	6.5	11.9	10.8
19	"	7	54.8	26.4	58.0	14.1	31.0	...	...	5.0	11.0	...	...	5.0	11.0	8.3
20	"	9	45.5	9.6	45.3	9.1	42.9	...	...	2.5	11.8	...	...	2.5	11.8	4.2

TABLE VI — LETTUCE

No.	DATE OF EXAMINATION	No. OF DAYS FROM THE COMMENCEMENT OF THE EXPERIMENT	TOTAL PIGMENTS	XANTHOPHYLL		UNIDENTIFIED PIGMENT OF BAND II		NEO- $\beta$ -CAROTENE U		$\beta$ -CAROTENE		NEO- $\beta$ -CAROTENE B		TOTAL ACTIVE PIGMENTS IN TERMS OF $\beta$ -CAROTENE		VITAMIN A POTENCY I.U./gm.
			$\mu\text{gm./gm.}$	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	$\mu\text{gm./gm.}$	%	
1	Feb.	3	187.3	113.6	60.6	7.1	3.8	10.0	5.3	53.7	28.7	2.9	1.6	57.65	30.8	96.1
2	"	5	201.6	129.6	64.3	9.0	4.5	12.5	6.2	43.0	21.4	7.5	3.7	49.87	24.7	83.1
3	"	14	187.1	113.1	60.4	8.1	4.3	8.0	4.2	54.7	29.3	3.2	1.7	58.30	31.2	97.2
4	"	15	170.7	101.3	59.3	10.1	5.9	10.0	5.9	45.6	26.7	3.7	2.2	49.95	29.3	83.3
5	"	17	191.9	123.8	64.5	9.6	5.0	9.6	5.0	40.8	21.3	8.1	4.2	47.25	24.6	73.8
6	"	22	168.4	104.6	62.1	10.0	5.9	7.6	4.5	42.1	25.0	4.1	2.4	46.05	27.3	76.8
7	"	21	159.7	96.6	60.6	8.1	5.0	8.8	5.5	40.0	25.0	6.2	3.9	45.30	28.4	75.5
8	"	25	162.1	108.1	66.7	2.8	1.7	4.8	3.0	44.1	27.2	2.3	1.4	46.40	28.6	77.4
9	"	24	146.0	94.7	64.9	6.4	4.4	7.0	4.0	35.3	20.3	4.4	6.9	37.45	25.6	62.4
10	"	28	174.0	113.0	65.1	6.5	3.7	7.0	4.0	35.3	20.3	12.0	6.9	43.05	24.7	71.8
11	Mar.	1	161.1	103.8	64.4	10.0	6.2	11.3	7.0	36.0	22.3	...	...	38.82	24.1	64.7
12	"	3	175.6	119.0	67.8	9.0	5.1	7.8	4.4	35.4	20.2	4.4	2.5	39.55	22.5	65.9
13	"	4	170.1	121.4	71.4	3.9	2.3	11.3	7.0	36.0	22.3	11.7	8.9	35.95	21.1	59.9
14	"	5	121.8	76.5	62.8	7.3	6.0	7.1	5.8	28.1	17.1	2.8	2.3	31.80	26.1	53.0
15	"	31	125.8	81.2	64.6	4.6	3.6	8.0	6.4	28.5	22.7	3.5	2.8	32.25	25.6	53.8
16	"	10	119.1	78.6	66.0	3.2	2.7	3.5	2.9	30.2	22.4	3.6	3.0	32.87	27.6	54.8
17	"	36	100.1	57.9	57.8	6.3	5.2	6.0	3.0	24.9	20.8	4.1	1.6	31.70	31.6	52.8
18	"	11	119.9	80.8	67.4	6.3	5.2	6.0	5.0	24.9	20.8	1.9	1.6	27.35	22.9	45.6
19	"	38	105.2	60.0	57.5	10.0	8.5	5.5	5.2	25.2	24.0	4.5	4.2	28.75	27.1	45.5
20	"	15	100.8	58.0	57.5	8.5	9.5	6.7	6.7	23.7	23.6	3.9	3.9	27.32	27.8	47.9
21	"	17	100.0	60.0	60.0	5.0	5.0	7.4	7.4	24.0	24.0	4.0	4.0	27.8	27.8	46.3
22	"	46	96.4	62.0	65.2	7.6	7.9	6.4	6.6	18.1	19.1	1.1	1.1	20.55	21.3	34.3

amount of acid-washed quartz sand was added and the tissues ground. The procedure followed for the complete extraction, isolation and estimation of the carotenoid pigments is described in an earlier paper<sup>20</sup>.

## Results

The chromatogram of all the leafy extracts showed the presence of five distinct bands. Details regarding the characterization of the bands has been described in an earlier paper<sup>21</sup>. Tables I-VI give the analytical data relating to the carotenoid pigments present in the leaves of cauliflower, fenugreek, carrot, rape seed, spinach and lettuce during the various stages of growth. The changes of xanthophyll and  $\beta$ -carotene as also in vitamin A activity are shown in Tables I-VI.

## Discussion

The leaves have been examined from their first appearance up to 46-56 days. The pigments identified in practically all the leaves were xanthophyll,  $\beta$ -carotene and its isomers, neo- $\beta$ -carotene B and neo- $\beta$ -carotene U, and an unidentified pigment. The two principal pigments, xanthophyll and  $\beta$ -carotene, steadily increased with the development of the leaves, reaching a maximum in 18-28 days after which they began to decline. This is particularly true in cauliflower, carrot leaves and spinach as may be seen from Tables I-VI.

The maxima is reached in 18 days for cauliflower, in 28 days for fenugreek, in 27 days for carrot leaves, in 18 days for rape seed and in 25 days for spinach.

The quantities of neo- $\beta$ -carotene B and neo- $\beta$ -carotene U present in the leaves showed slight variations.

The unidentified pigment of band II is present in relatively small amounts, 3-12 per cent, and shows very slight variations during the development of the leaves. In the later stages, however, when the leaves were beginning to turn yellow, the quantity of this pigment increased to about 32.8 per cent in cauliflower and 42.9 per cent in spinach.

Lettuce, however, showed a different behaviour. The examination of the leaves of this plant were started with the outer green leaves which had attained a relatively large size. The estimation of the pigments was carried out for a period of 7 weeks at bi-weekly intervals and a progressive fall was obtained in all the pigments. The rate

of fall in both  $\beta$ -carotene and xanthophyll varied from 10-30 per cent per week.

## Summary

Variations in the carotenoid content of the leaves of six vegetables during growth were studied. The materials examined are cauliflower, fenugreek, carrot leaves, rape seed leaves, spinach and lettuce.

In all the leafy vegetables except lettuce, both xanthophyll and  $\beta$ -carotene showed a steady increase during the early stages of growth, after which there was a decline. The values for neo- $\beta$ -carotene B and neo- $\beta$ -carotene U remain unaltered during growth.

Lettuce showed a decrease both in xanthophyll and  $\beta$ -carotene values throughout the period of experiment. It is possible that in this species maximum values are reached very quickly.

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# Influence of Nucleic Acid on the Action of Streptomycin on Certain Pathogens

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IN previous papers<sup>1-6</sup> we have drawn attention to the importance of nucleic acids as agents tied up intimately with the bacteriostatic effect of penicillin. The experiments had clearly shown that penicillin brings about the bacteriostasis of pathogens by interfering with those phases in the metabolic and reproductory processes in which nucleic acids play important rôles either as metabolites helping cell division or as respiratory catalysts or as both. Parallel experiments have been carried out with streptomycin which has now become an equally important chemotherapeutic agent active in both Gram-positive and Gram-negative organisms as also on acid-fast organisms like *M. tuberculosis*. The results reported in this paper relate to bacteriostatic effects of streptomycin.

L. P. Garrod<sup>7</sup> has stressed that "the therapeutic effects of streptomycin are due to bacteriocidal rather than bacteriostatic

action". Our experiments show that this is so only when high concentrations of streptomycin are used.

## Experimental

The experimental details in these studies are the same as those described in previous papers (loc. cit.). First, the minimum inhibiting concentration of streptomycin hydrochloride dissolved in phosphate buffer for each test organism was determined by serial dilution method in nutrient broth of pH 7.6. 24-hour old broth cultures of the test organism were employed for each experiment in sterile tubes, in duplicate, containing a total volume of 10 c.c. including nutrient broth, streptomycin solution in the requisite amounts to give the final concentrations, and nucleic acid. (A sample of yeast nucleic acid from B.D.H. and streptomycin hydrochloride from Messrs Squibb's were used.) The cultures were then incubated,

TABLE I—EFFECT OF NUCLEIC ACID ON THE INHIBITORY ACTIVITY OF STREPTOMYCIN HYDROCHLORIDE

ORGANISM	CONCN. OF STREPTOMYCIN ALONE	GROWTH IN BROTH	CONCN. OF NUCLEIC ACID ALONE	GROWTH IN BROTH	NUCLEIC ACID + STREPTOMYCIN	GROWTH IN BROTH	CONTROL IN BROTH
<i>St. aureus</i>	$\frac{1}{500,000}$	...	$\frac{1}{1,000}$	+ +	$\frac{1}{1,000}$	$\frac{1}{500,000}$	+
	$\frac{1}{750,000}$	...				$\frac{1}{750,000}$	+
	$\frac{1}{1,000,000}$	...				$\frac{1}{1,000,000}$	+
	$\frac{1}{1,000,000}$	...	$\frac{1}{500}$	+ +	$\frac{1}{500} +$	$\frac{1}{500,000}$	++
	$\frac{1}{2,000,000}$	+				$\frac{1}{750,000}$	++
						$\frac{1}{1,000,000}$	++
<i>Esch. coli.</i>	$\frac{1}{500,000}$	...	$\frac{1}{1,000}$	+ +	$\frac{1}{1,000} +$	$\frac{1}{500,000}$	+
	$\frac{1}{750,000}$	...				$\frac{1}{750,000}$	++
	$\frac{1}{1,000,000}$	+				$\frac{1}{1,000,000}$	++
			$\frac{1}{500}$	+ +	$\frac{1}{500} +$	$\frac{1}{500,000}$	+
						$\frac{1}{750,000}$	++
						$\frac{1}{1,000,000}$	++

and after 24 hours the growth was observed by visual turbidity or its absence. The results are given in Table I.

It may be seen that nucleic acid is able to inactivate the bacteriostatic effect of streptomycin on both *St. aureus* and *Esch. coli*. In the case of penicillin this was not so, and penicillin action was reversible in the case of *St. aureus* and not in the case of *Esch. coli*. Streptomycin thus appears to interfere with the nucleic acid metabolism of both Gram-positive and Gram-negative

organisms. The higher the concentration of nucleic acid added, the more complete is the inactivation of streptomycin.

These results make it clear that nucleic acid renders viable both Gram-positive and Gram-negative organisms which in the presence of minimum inhibitory concentrations of streptomycin became non-viable.

In Table III is shown the absence of any inhibitory or reversing effect of magnesium ribonucleate on streptomycin action on Gram-

TABLE II—REVERSIBILITY OF STREPTOMYCIN INHIBITION BY NUCLEIC ACID

ORGANISM	CONCN. OF STREPTOMYCIN ALONE	GROWTH IN BROTH	GROWTH IN BROTH WHEN NUCLEIC ACID IS ADDED AFTER:		CONTROL IN BROTH
			3 hr.	6 hr.	
<i>St. aureus</i>	$\frac{1}{500,000}$	...	+	+	
	$\frac{1}{750,000}$	...	...	...	++
	$\frac{1}{1,000,000}$	...	++	++	
	$\frac{1}{1,000,000}$	...	++	++	
<i>Esch. coli.</i>	$\frac{1}{500,000}$	...	+	+	
	$\frac{1}{750,000}$	...	+	+	++
	$\frac{1}{1,000,000}$	+	++	++	
	$\frac{1}{1,000,000}$	+	++	++	

TABLE III—ABSENCE OF ANY EFFECT OF MAGNESIUM RIBONUCLEATE ON GRAM-POSITIVE & GRAM-NEGATIVE ORGANISMS

ORGANISM	CONCN. OF STREPTOMYCIN ALONE	GROWTH IN BROTH	C.C. OF MG. RIBONUCLEATE ALONE (1/1,200)	GROWTH IN BROTH	C.C. OF MG. RIBONUCLEATE + STREPTOMYCIN	GROWTH IN BROTH	CONTROL IN BROTH
<i>St. aureus</i>	$\frac{1}{500,000}$	...	1	++	1 + $\frac{1}{500,000}$	...	
	$\frac{1}{750,000}$	...			1 + $\frac{1}{750,000}$	...	
	$\frac{1}{1,000,000}$	...	2	++	2 + $\frac{1}{1,000,000}$	...	++
	$\frac{1}{1,000,000}$	...	2	++	2 + $\frac{1}{1,000,000}$	...	
<i>Esch. coli.</i>	$\frac{1}{500,000}$	...	1	++	1 + $\frac{1}{500,000}$	...	
	$\frac{1}{750,000}$	...			1 + $\frac{1}{750,000}$	+	
	$\frac{1}{1,000,000}$	+	2	++	2 + $\frac{1}{1,000,000}$	+	++
	$\frac{1}{1,000,000}$	+	2	++	2 + $\frac{1}{1,000,000}$	+	

positive and Gram-negative organisms. The absence of any influence of magnesium ions on the growth of *St. aureus* and *Esch. coli* on the inhibitory concentrations of streptomycin is shown in Table IV. Streptomycin is inhibitory to both the organism and added

magnesium ions seem to have no effect in reducing the minimum bacteriostatic concentration of the antibiotic. In the case of penicillin the minimum inhibitory requirement came down under the same condition by 50 per cent.

TABLE IV—INFLUENCE OF MAGNESIUM IONS ON THE GROWTH OF *ST. AUREUS* & *ESCH. COLI* IN THE PRESENCE OF STREPTOMYCIN

ORGANISM	CONCN. OF STREPTOMYCIN ALONE	GROWTH IN BROTH	Mg. IONS (% Mg SO <sub>4</sub> ) ALONE	GROWTH IN BROTH	Mg. IONS (% MgSO <sub>4</sub> ) + STREPTOMYCIN	GROWTH IN BROTH	CONTROL IN BROTH
<i>St. aureus</i>	1	...	0.1	++	0.1 +	1	+
	500,000	...				500,000	+
	1	...				1,000,000	++
	750,000	...	0.2	++	0.2 +	1	++
	1,000,000	...				500,000	+
	2,000,000	+				1	+
<i>Esch. coli</i>	1	...	0.1	++	0.1 +	1	+
	500,000	...				500,000	+
	1	...				750,000	++
	750,000	...	0.2	++	0.2 +	1,000,000	++
	1,000,000	+				1	+
						500,000	+

TABLE V—EFFECT OF NUCLEIC ACID ON THE INHIBITORY ACTIVITY OF STREPTOMYCIN ON *E. TYPHOSUM*

ORGANISM	CONCN. OF STREPTOMYCIN ALONE	GROWTH IN BROTH	NUCLEIC ACID ALONE	GROWTH IN BROTH	NUCLEIC ACID " + STREPTOMYCIN	GROWTH IN BROTH	CONTROL IN BROTH
<i>E. typhosum</i>	1	...	0.1	++	0.1 +	1	+
	250,000	...				250,000	+
	1	...				500,000	++
	500,000	...	0.2	++	0.2 +	1,000,000	++
	1,000,000	+				1	++
						250,000	++

TABLE VI—REVERSIBILITY OF STREPTOMYCIN ACTION IN *E. TYPHOSUM* BY ADDED NUCLEIC ACID

ORGANISM	CONC. OF STREPTOMYCIN ALONE	NUCLEIC ACID ADDED AFTER		CONTROL
		3 hr.	6 hr.	
<i>E. typhosum</i>	1	++	++	
	250,000	++	+	++
	1			



In Tables V and VI are presented the effects of nucleic acid on the inhibitory action of streptomycin on a non-penicillinase producing Gram-negative organism, viz. *E. typhosus* as well as the reversibility of the inhibitory action.

It may be seen that in the case of this organism, arrest of growth by streptomycin is prevented by nucleic acid. The inhibitory action is also reversible.

### Discussion

In the experiments reported above, streptomycin was used in concentrations very near to the bacteriostatic range such that its action never caused permanent injury to the cells. The results show that there are significant differences between streptomycin and penicillin in the manner in which they exert their bacteriostatic action. This is to be expected from the difference in the chemical nature and properties of the two antibiotic agents. While nucleic acid was able to render viable only Gram-positive organisms and not Gram-negative organisms under the influence of penicillin, it could render viable both Gram-positive and Gram-negative organisms under the influence of streptomycin. It may thus be assumed that the mechanisms influenced by streptomycin bacteriostasis are similar in the case of all pathogens irrespective of their Gram-staining reactions. This is different in the case of penicillin as was shown previously<sup>1</sup>. Again magnesium ribonucleate was found to be ineffective in inhibiting streptomycin bacteriostasis as well as in reversing streptomycin inhibition of either Gram-positive or Gram-negative organisms while it could reverse the penicillin inhibition of Gram-negative organisms.

Another noteworthy difference in the action of the two antibiotics is the fact that when pathogens like *St. aureus*, *Esch. coli*, *E. typhosum* were treated with sub-bacteriostatic concentrations of streptomycin and their Gram reaction tested after short periods, it was found that, quite unlike the observations made in the case of penicillin, all organisms retained their original morphological as well as Gram-staining characteristics. Again, while magnesium ions play an important part in bringing down the minimum inhibiting concentration of penicillin for organisms like *Esch. coli*, *B. dysenteriae*, etc., streptomycin inhibition was unaffected. The mechanism of the effects brought about by nucleic acid in the bac-

teriostatic action of the antibiotic on the respective pathogens is not clear. It is not improbable that streptomycin action being optimum in an alkaline medium, added nucleic acid tilts the pH to the acidic side, consequently inactivating the antibiotic agent. This, however, is not so in the case of penicillin<sup>6</sup>.

### Summary

1. Nucleic acid reverses the bacteriostatic action of streptomycin on common pathogens such as *St. aureus*, *Esch. coli*, etc. The higher the concentration of the nucleic acid, the more complete the inactivating effect on the antibiotic.

2. Nucleic acid renders viable both Gram-positive *St. aureus* and Gram-negative *Esch. coli* which were in contact with bacteriostatic concentrations of streptomycin.

3. Magnesium ribonucleate is ineffective in inhibiting streptomycin bacteriostasis as well as in reversing the streptomycin inhibition of both Gram-positive and Gram-negative organisms.

4. Pathogens like *St. aureus*, *Esch. coli* and *E. typhosum* treated with sub-bacteriostatic concentrations of streptomycin retained their original morphological and Gram-staining characteristics quite unlike that with penicillin.

5. Magnesium ions, which play an important part in bringing down the minimum inhibiting concentration of penicillin for certain Gram-negative organisms, do not have any effect on streptomycin inhibition.

6. The differences in the manner in which the bacteriostatic effects are achieved by the two antibiotic agents, penicillin and streptomycin, have been pointed out.

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# Coir Fibre — Structure & Identification of Damage

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**C**OIR occupies an important position in the agricultural economy of this country and finds wide application in industry. Coir products are used under diverse conditions which often render them liable to deterioration. Further, during storage, they are often damaged in accidental contact with acids, alkalies, oxidants and other agencies. It is of considerable importance to distinguish the varying types of deterioration, as this will enable one to assess the serviceability of the material under different conditions, and also to adopt suitable measures against deterioration.

With cotton and other cellulose fibres, a number of well-known chemical methods<sup>1</sup> are available for the identification of different types of tendering. These methods, however, are not applicable to coir, in which the constituent material is cellulose-lignin complex and not cellulose, and no specific methods were available hitherto for use in relation to this material. The present paper gives a brief account of the structure of coir fibre and methods for distinguishing varying types of damage to this fibre.

## Structure of Coir Fibre

The coir fibres are embedded in a mass of highly elastic cork-like parenchymatous tissue of the husk (FIG. 1). When fresh, they are of light colour, soft and filled with tannin and sugar. On drying, some of the organic substances become insoluble and impart a permanent dark colour to the fibres and the surrounding parenchymatous cells. The length, diameter and uniformity of fibres vary in the different regions of the coconut husk from which they are derived. Except for some minor differences in colour, texture and fineness, samples from different parts of India do not show any marked variation in physical or chemical properties.

The fibres are derived from leaf trace bundles and are composed of a rudimentary protoxylem enclosed by a thick sclerenchymatous sheath. The protoxylem elements are usually composed of a few annular and

spiral cells. In the thick fibres of the mesocarp, the protoxylem region is comparatively large. During retting, the protoxylem disintegrates, resulting in a hollow central zone which serves as a nucleus for any subsequent microbial deterioration. Sclerenchymatous cells are long, narrow, pointed at ends and due to sliding growth become interlocked in dove-tail fashion thereby adding to the elasticity and tensile strength of the fibres. The cell wall is thick and the lumen narrow, containing only air. The secondary thickening is uniform and traversed by numerous simple and some funnel-shaped pits (FIG. 2). In addition to secondary thickening, cells exhibit mostly delicate reticulate and a few annular or spiral thickenings. The thickening as well as the length of sclerenchymatous cells vary

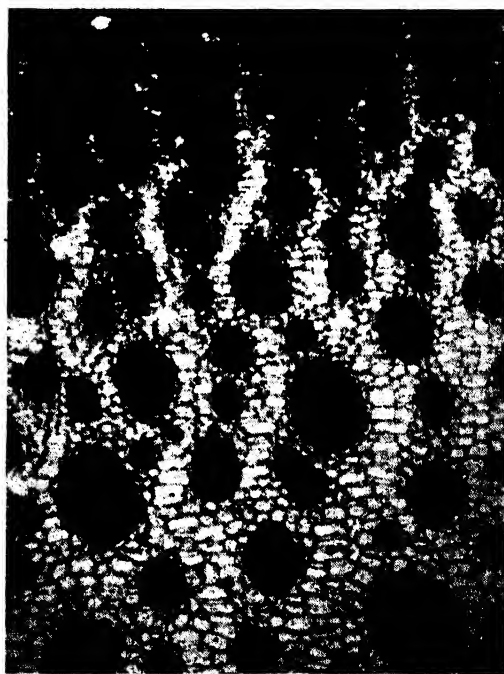
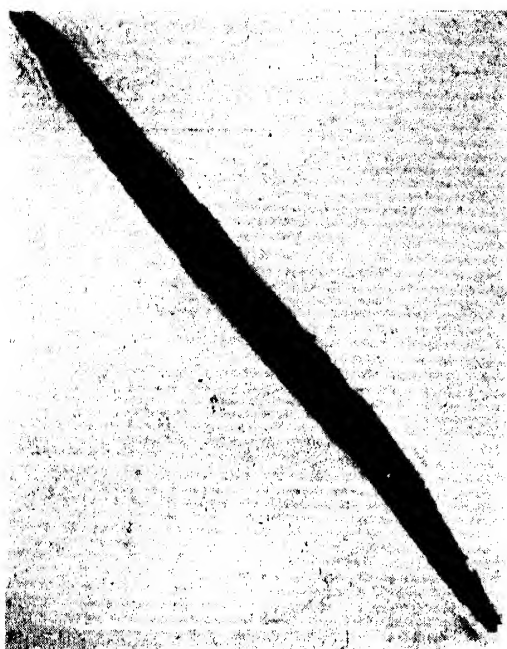


FIG. 1 — T.S. OF COCONUT HUSK SHOWING THE DISTRIBUTION OF DIFFERENT TYPES OF FIBRES  $\times 50$ .

FIG. 2 — A SINGLE COIR CELL  $\times 200$ .

considerably even in the different regions of the same fibre. On maceration or treatment with suitable reagents, small lens-shaped silicified structures called stegmata cells may be seen on the surface of a fibre (FIG. 3).

The cell wall is composed mainly of cellulose and lignin. The failure of previous investigators to recognize cellulose<sup>2,3</sup> in coir by micro-chemical test may be due to the fact that when the primary cuticle is intact, the cellulose reaction takes place only after 10-15 minutes. If the secondary wall is exposed by chemical or mechanical methods, the reaction takes place immediately.

#### Coir Deterioration & Identification

*Types of Deterioration* — Two agencies are mainly responsible for the deterioration of coir. These are: (1) chemical, and (2) biological. Chemical tendering usually takes place as a result of accidental contact with acids, alkalies and other chemicals during storage or transit. Another type of chemical tendering appears when the material is subjected to weathering. This is apparently a case of oxidation catalysed by sunlight. Biological tendering is due to micro-organisms and takes place especially when coir is in contact with soil, or is con-

taminated with material capable of acting as nutrients for micro-organisms.

The methods of identifying tendered fibres are as follows:

*Distinction between Normal & Tendered Fibres* — Materials are teased on a slide, treated for 3 min. with 1 per cent safranin solution in 50 per cent alcohol, washed and mounted in water on a slide.

*Normal Fibres* — Stain pink or violet.

*Tendered Fibres* — Stain deep red.

*Chemical Tendering* — Fibres tendered chemically do not show any morphological or histological differences from normal fibres unless they are treated with a swelling reagent and suitably stained. For this purpose zinc chloridide solution is used. The reagent is prepared as follows:

A		
Zinc chloride	..	20 gr.
Water	..	10 c.c.
B		
Potassium iodide	..	2.0 gr.
Iodine	..	0.1 gr.
Water	..	5 c.c.

Solutions A and B are mixed, decanted after 24 hr. and kept in a dark-coloured reagent bottle.

Fibres are treated with a drop or two of the reagent, gently teased on a slide and

FIG. 3 — A CELL SHOWING STEGMATA CELLS  $\times 200$ .

heated over a steam bath to produce a high swelling. The period for which a slide is to be heated varies from 1-9 minutes depend-

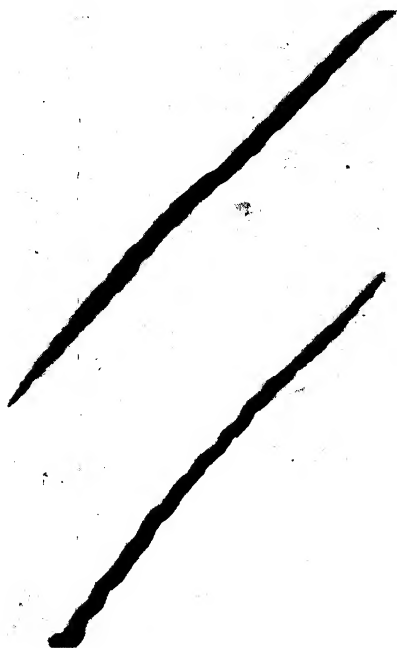


FIG. 4 — CELL TENDERED BY HYDROCHLORIC ACID  $\times 200$ .



FIG. 5 — CELL TENDERED BY NITRIC ACID  $\times 100$ .

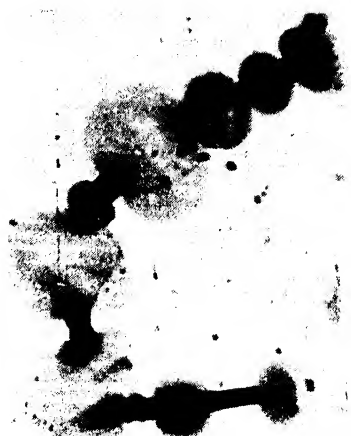


FIG. 6 — CELL TENDERED BY SULPHURIC ACID  $\times 100$ .



FIG. 7 — CELL TENDERED BY ALKALI  $\times 200$ .

ing on the type and the degree of deterioration. Over-heating, however, completely disintegrates the fibres. In practice, the slide



FIG. 8 — T.S. OF COIR FIBRE AFTER SOIL BURIAL TEST SHOWING THE DISINTEGRATION OF THE INTERIOR CELLS  $\times 200$ .

is examined under the microscope at frequent intervals for the characteristic features of different types of tendering (see below). The slide is then removed, allowed to cool and excess of reagent removed with a filter paper. A drop or two of fresh reagent is added and the fibre examined under a microscope.

**HCl-tendered Fibres** — Cells show regular wavy curling throughout the entire length with cuticle remaining intact (FIG. 4).

**HNO<sub>3</sub>-tendered Fibres** — Cuticle is ruptured at intervals accompanied by slight swelling and sharp segmentation (FIG. 5). Some of the sclerenchymatous cells with thick secondary wall show slight curling at ends only.

**H<sub>2</sub>SO<sub>4</sub>-tendered Fibres** — The cells swell considerably forming regular bubbles with sharp constrictions in between, resembling a beaded string (FIG. 6). Some short cells with thick secondary wall show slight curling and occasional segmentation.

**NaOH or KOH-tendered Fibres** — Curling as noticed in acid tendering is absent. Cells show formation of bubbles as when tendered by sulphuric acid. The distinction, however, lies in the presence of the central lumen inside the bubbles (FIG. 7).

**Fibres Tendered by Oxidation** (e.g. with bleaching powder, hydrogen peroxide, through exposure, etc.) — On treatment of oxidized fibres with the reagent, the entire secondary wall immediately turns deep blue, whereas normal fibres under similar conditions show only slight discontinuous blue colouration after 10-15 min. Fibres treated with hydrogen peroxide show, in addition, the formation of bubbles as in alkali-tendered cells, but these bubbles are small and distantly spaced with fairly long intact fibres in between.

The characteristic transformations described above are brought about by the different chemicals employed either in strong or in weak solutions. The period of reaction depends on the strength of the solution, and it has been found that the distinctive reactions with acids and alkalis can be detected even with 1 per cent concentration of the chemicals. In Table I are set out the details of the treatments.

TABLE I

REAGENT	CONCENTRATION % wt. Vol.	FIBRE	PERIOD OF REAC- TION	REACTION TEMP. °C.
Nitric acid	60	Fibres as such	24 hr.	28
	50	"	24 hr.	28
	1.0	Cut, crushed and macerated	30 min.	100
Sulphuric acid	98	Fibres as such	10 sec.	28
	85	"	8 hr.	28
	85	"	10 min.	100
	2	Cut, crushed, and macerated	30 min.	100
	1	"	1 hr.	100
Hydrochloric acid	34	Fibres as such	30 min.	28
	20	"	96 hr.	28
	0.5	Cut, crushed and macerated	30 min.	100
Sodium hydroxide	50	Fibres as such	45 min.	28
	30	"	24 hr.	28
	1	Cut, crushed and macerated	30 min.	100
Hydrogen peroxide	12 volumes	Fibres as such	24 hr.	28
Bleaching powder (avail. chlorine 22.3%)	5% suspension in water	Fibres as such	10 min	100

### Biological Tendering

The zinc chloriodide technique did not show any characteristic feature in samples tendered by biological agencies. It has been found, however, that such tendering can be distinguished by direct microscopic examination and staining of the material. The manner of attack as well as the nature

of damage differ in respect of bacteria and fungi. The bacterial attack appears at first in the disintegrated protoxylem of the fibre and gradually proceeds towards the periphery, corroding the sclerenchymatous cells so that, in a cross-section, the fibre presents a number of irregular indented hollow zones while the outer surface shows hardly any deterioration (FIG. 8). A further distinguishing feature of bacterial damage is the presence of numerous minute indentations on the surface of the fibre cells. In the case of fungi, surface indentations or disintegration of the central zone of the fibre is absent. The lumen of the fibre cells do not show the presence of hyphae which usually entwine round the fibre and degradation is noticed in regions immediately in contact with hyphae. The presence of some of the remnants of the protoxylem elements and adhering cork cells appears to add to the susceptibility of the fibres to any subsequent microbial attack.

#### Summary

No specific method has been hitherto recorded for identification of damages in coir. The

present paper describes a procedure for distinguishing different types of chemical and biological deterioration of this material.

#### Acknowledgements

The authors desire to record their grateful thanks to Dr. T. S. Subramanian for his interest in the work and to the Deputy Chief of the General Staff (W. & E.) and the Director of Technical Development, Army Headquarters (India) for kind permission to publish the paper. Thanks are also due to the Department of Industries and Commerce, Government of Cochin, Chamber of Commerce, Travancore; Messrs S.K.V. Industries, Paraveer; and Messrs Pierce Leslie & Co., Travancore for the supply of coir samples.

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## Letters to the Editor

### THE ENHANCEMENT OF PENICILLIN EFFECTIVENESS *IN VIVO* BY TRACES OF MAGNESIUM

IN EARLIER PUBLICATIONS<sup>1,2</sup> WE HAVE shown that magnesium ions have a pronounced influence on the bacteriostatic effects of penicillin on Gram-negative organisms like *Esch. coli*, *in vitro*. Indeed, similar effectiveness on penicillin action against *Eberth. typhosa* by the addition of small amounts of cobalt *in vivo* by injection in adult mice was observed by Pratt *et al*<sup>3</sup>. In view of the increasing interest to make

penicillin more versatile and efficient, specially in the case of Gram-negative pathogens, we studied the effect of using a mixture of penicillin and magnesium as  $MgSO_4$  in *Esch. coli* count of the intestinal tracts of experimental animals like mice and rats.

Experiments were conducted by administering magnesium sulphate (0.1 per cent) orally and penicillin by subcutaneous injection and also by oral administration of the mixture of magnesium sulphate and penicillin dispersed in peanut oil. The results obtained are recorded in Tables I and II.

TABLE I — *ESCH. COLI* COUNTS IN THE FAECES OF MICE BEFORE & AFTER PENICILLIN INJECTION

MOUSE No.	COUNT BEFORE TREATMENT			COUNT AFTER TREATMENT		FINAL COUNT INITIAL COUNT (1st day) × 100
	1st day	2nd day	3rd day	Penicillin alone (200 units)	Penicillin + $MgSO_4$	
1	420	360	502	304	...	72
2	313	400	280	208	...	66
3	606	420	625	...	200	53
4	353	375	204	...	148	42

TABLE II—*ESCH. COLI* COUNTS IN THE FAECES OF RATS BEFORE & AFTER ORAL ADMINISTRATION OF PENICILLIN

RAT No.	COUNT BEFORE TREATMENT			COUNT AFTER TREATMENT						FINAL COUNT INITIAL COUNT (1st day) $\times 100$
	1st day	2nd day	3rd day	PENICILLIN ALONE			PENICILLIN+MgSO <sub>4</sub>			
				1st	2nd	3rd	1st	2nd	3rd	
1	150	108	156	62	81	85	...	...	...	41
2	1140	990	1026	538	760	692	...	...	...	49
3	340	252	423	...	...	...	106	68	135	31
4	738	640	530	...	...	...	228	132	192	31
1	1320	835	1086	382	321	378	...	...	...	30
2	1500	2490	2645	527	218	320	...	...	...	35
3	2690	1950	2550	...	...	...	598	41	110	22
4	2800	2250	2655	...	...	...	650	192	264	23

In all cases the dosage of penicillin used was arbitrarily taken as calculated from preliminary experiments.

It may be concluded from the results that *in vivo* appropriate doses of magnesium sulphate along with penicillin would be effective in increasing and prolonging the effect of penicillin. Magnesium sulphate alone in the same concentration, and even slightly higher concentration, had no inhibitory effect. These results also show that it should be possible to evolve conditions when penicillin with certain stabilizing adjuncts like magnesium sulphate can be orally administered with as much advantage as can be obtained. Pratt *et al.*'s as well as our experiments suggested the possibility of using certain cations like magnesium and cobalt to increase the efficiency of penicillin to treat certain Gram-negative bacterial infections.

Further studies on other organisms are in progress.

Our thanks are due to Prof. V. Subrahmanyam and Major K. P. Menon for their kind interest in this work, and to the *Council of Scientific & Industrial Research*, New Delhi, for the financial help. We also thank *Messrs Eli Lilly & Co.*, U.S.A. for the supply of penicillin used in these experiments.

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#### PURIFICATION OF CRUDE CALCIUM LACTATE

A PROCESS FOR THE PURIFICATION OF CRUDE calcium lactate solutions obtained by the fermentation of cane molasses has been worked out and adopted on a commercial scale. The crude product obtained from molasses contains many impurities, and the usual methods<sup>1-10</sup> to remove them have met with little success. The process now worked out consists in treating a concentrated solution of crude calcium lactate with the calculated quantity of commercial ferrous sulphate in the hot. On standing for a few hours, a heavy precipitate of ferrous lactate and calcium sulphate settles down. The precipitate is separated on the centrifuge and washed, then suspended in boiling water, and milk of lime added till alkaline. The pale-yellow coloured solution containing all the calcium lactate is filtered off, boiled with activated carbon (0.5 per cent on the weight of calcium lactate) and filtered. The final solution is concentrated (sp. gr. 1.07 at 30°C.) and seeded with calcium lactate, when crystals of calcium lactate separated out. The slurry is centrifuged and the calcium lactate dried in the usual manner.

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Rampur

October 25, 1948

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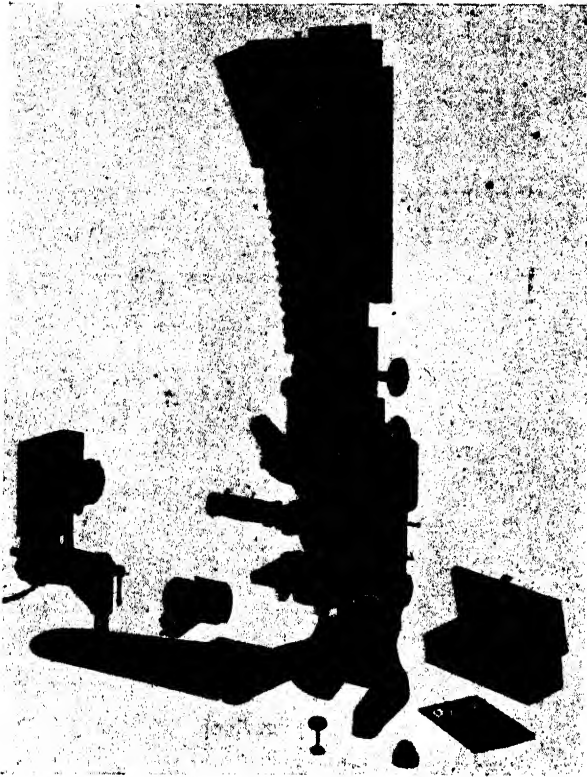
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# A Study of Coarse Filtering Media for Portable Producer-Gas Plant Filters\*

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TOWARDS the close of 1941 a search for the alternative motor fuels was made in this country due to petrol shortage. One of the means to overcome such a difficulty was the development of producer-gas plants suitable for use on transport vehicles. At the time of conversion of the existing types of transport vehicles to producer gas, the Indian manufacturers had nothing to guide them, because a scientific study on the subject had not been undertaken earlier. The manufacturers of the producer-gas plants showed a great ingenuity in the design of the filters by using the various available fibrous materials for gas filtration. During the past seven years, however, an increasing amount of attention has been paid to the scientific development of transport using producer gas as a source of power, together with the design, manufacture and testing of mobile producer-gas plants.

Abrasive dust, tarry matter and corrosive gases are present in the gas. The life of an engine running on producer gas mainly depends on the extent to which these impurities have been got rid of from the gas as finally supplied to the engine. The satisfactory performance, on the other hand, demands free and ample flow of clean gas with the least pressure drop in the delivery system. This pressure drop mainly takes place in the filtration system. To meet the situation, two different types of filters, namely wet and dry, have been developed and employed in various countries.

In India the general practice followed for gas filtration is to pass it through a

series of dry filters, generally three in number. Fibrous materials and textile fabrics are made use of in these filters. After a careful study of the available textile fabrics in this country, the material most suited for use in the final stage of gas filtration<sup>1</sup> has been previously recommended and a suitable filter design has been suggested. It was, therefore, felt desirable to investigate the various filtering media available for use in the first two stages of gas filtration. A knowledge of the flow characteristics of gas through these media with respect to the pressure drop, depth of packing, packing density and the influence of these factors on the size of particles passing through the filter bed are absolutely necessary for the development and design of these filters. Investigations relating to these factors are described in the present paper.

In the filters of a portable producer-gas plant the materials most frequently used as coarse filtering media are cotton waste, jute, sisal, coir and *munj*. While selecting these materials, the various factors to be considered are:

- (i) cheapness;
- (ii) easy availability;
- (iii) number of times it is fit for re-use in the filter after cleaning or dusting; and
- (iv) compressibility.

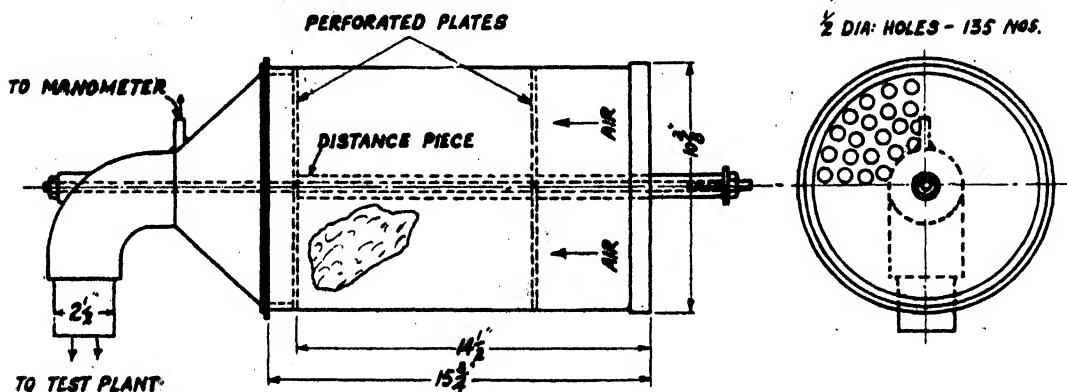
The selected materials were procured from the market. Before undertaking any test, these materials were carefully cleaned by hand-picking of any foreign materials present and dried in the sun. General description, price, place of origin and the

\*Paper read before the Engineering and Metallurgical Section of the 36th Indian Science Congress Session, Allahabad.

TABLE I

No.	MATERIAL	ORIGIN	COLOUR	PRICE PER LB., ANNAS	AVERAGE FIBRE DIA., IN MICRONS	CHARACTERISTICS OF THE MATERIAL
1.	Cotton waste	Mill waste	White	3	428*	Soft, easy to pack, sufficient control on quantity and sizes of particles passing, very difficult to clean
2.	Jute	Bengal	Light yellow	4	73	Soft, easy to pack, considerable control on quantity and sizes of particles passing, easy to clean
3.	Sisal	Bihar and Orissa	White	...	158	Hard, easy to pack, less control on quantity and sizes of particles passing, easy to clean
4.	Coir	South India	Brown	5	360	Hard, brittle and difficult to pack, much less control on quantity and sizes of particles passing, very easy to clean
5.	Munj	Punjab	Light yellow	4	228	Very hard, brittle, very difficult to pack, little control on quantity and sizes of particles passing, very easy to clean

\* The diameter of yarn is given in this case.



### LINEAR FILTER

FIG. 1

behaviour of these materials have been summarized in Table I.

As indicated in Table I, all these materials are available in this country and the cost per lb. of the material is not more than five annas in each case. The common practice followed in cleaning them is by dusting or beating the material with a stick. Some of these materials are easy to clean while others are not. This negative property of the material leads to frequent renewals and, therefore, increases the cost of maintenance. Some of them can be compressed more easily than others by keeping the packing space constant. They are generally soft, can be easily handled, give uniform packing, require less filtering area and have sufficient control on the quantity and sizes of dust particles passing through the pores created by the packing material. The main requirements

of a good filtering material generally are that it should allow the least quantity of dust particles to pass through the filter bed and, at the same time, allow a sufficient quantity of clean gas to pass through a given filtering area with the least pressure drop.

The apparatus employed during this investigation was the producer-gas bench-testing unit<sup>2</sup> installed in these laboratories. A 29.4 h.p. Chevrolet engine was made to act as a pump of the positive displacement type and was coupled through a Chevrolet standard gear box to a 25 h.p. 3-phase induction motor. The material under investigation was packed in a filter, which was connected at the end of the intake pipe of the testing unit. Air was drawn through the filter at various rates of flow and was discharged to the atmosphere through a

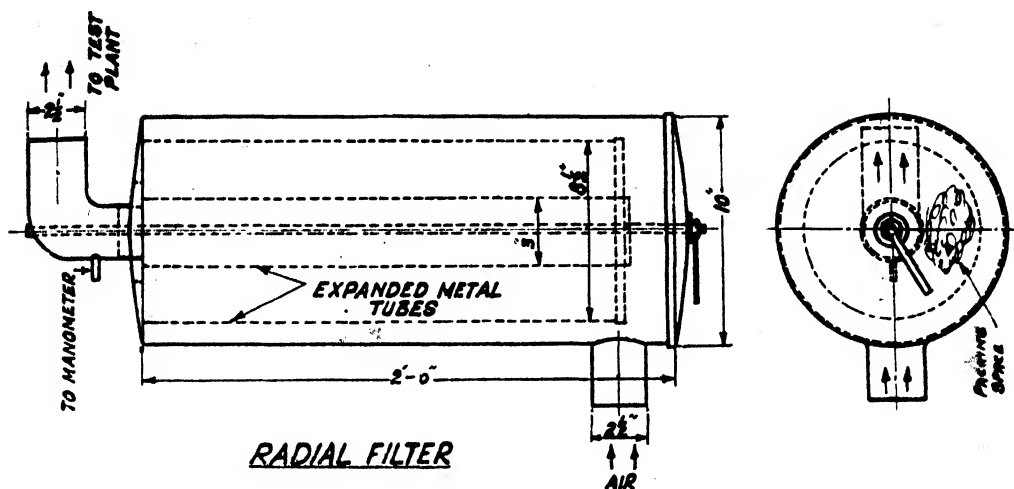


FIG. 2

calibrated orifice meter via an anti-pulsating tank. The atmospheric pressure and temperature prevailing at the time of test were recorded. The flow was corrected to 15°C. and 762 mm. of Hg in every case.

Two different types of filters, namely linear and radial, were used for these tests. They are named according to the flow of gas inside these filters. A linear filter is diagrammatically illustrated in Fig. 1. It consists of a cylindrical shell of 10 $\frac{3}{8}$ " diameter by 14 $\frac{1}{2}$ " long with two M.S. perforated circular plates. The plate at the gas outlet end was fixed in position, whereas the position of the plate at the gas inlet end could be adjusted according to the depth of packing required. The pressure difference across the filter bed was measured with the help

of a water manometer. An arrangement for connecting it across the filter was provided.

Each of these materials was packed and tested for pressure drop at maximum packing density, which could be attained by hand packing. The depths of packing chosen for experimental purposes were 2", 5", 8" and 11". The pressure drop across the filter bed for the various rates of air flow at different depths of packing at maximum packing density was determined. These values were also checked for decreasing rates of flow of air. These results in the case of cotton

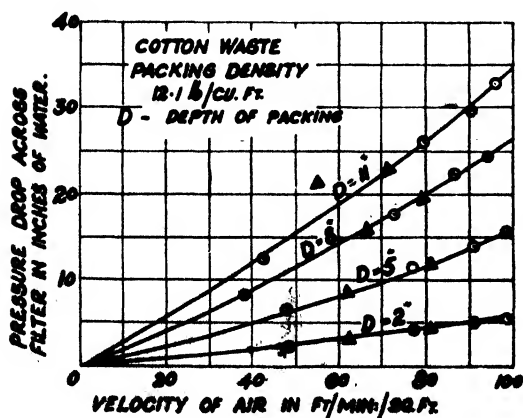


FIG. 3 — PRESSURE DROP VS. VELOCITY OF AIR FOR COTTON WASTE.

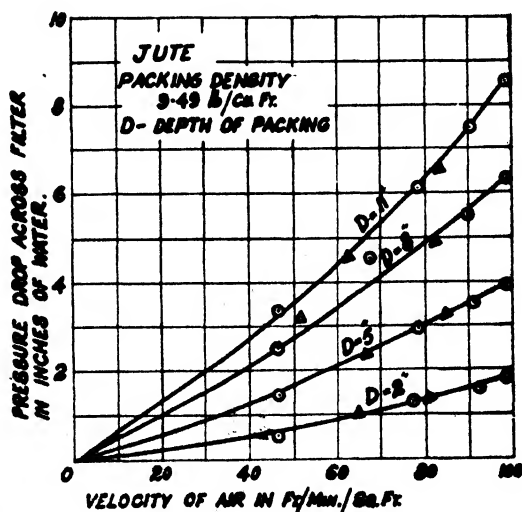


FIG. 4 — PRESSURE DROP VS. VELOCITY OF AIR FOR JUTE.

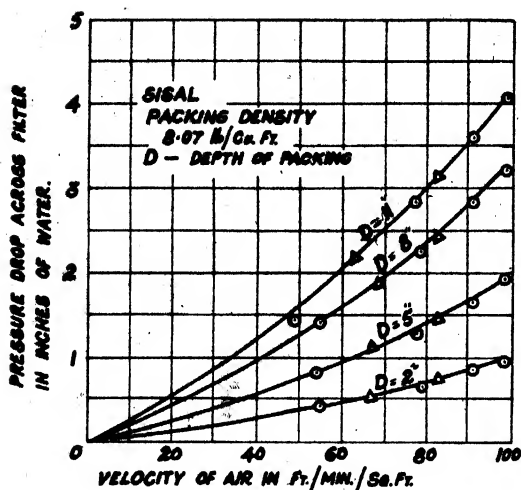


FIG. 5 — PRESSURE DROP VS. VELOCITY OF AIR FOR SISAL

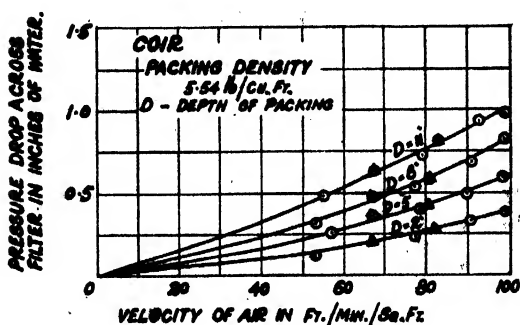


FIG. 6 — PRESSURE DROP VS. VELOCITY OF AIR FOR COIR.

waste, jute, sisal, coir and *munj* have been graphically represented in Figs. 3, 4, 5, 6 and 7 respectively. The pressure drop for the empty filter for the various rates of air velocity was also determined. In Fig. 8 are given the curves showing the relationship between pressure drop and depth of packing at only maximum packing density for the five materials studied presently.

At the end of every set of pressure drop test, the filter unit was connected at the end of the conical filter unit<sup>3</sup>. It consisted of two conical funnels of about 21" diameter with provision for clamping a 20" filter paper between them. In the filter unit a standard filter paper<sup>3</sup> was clamped so as to arrest the dust particles passing through the filter paper. Air at the rate of 50 cubic feet per minute<sup>4</sup>, which represents the full rate of gas flow for a heavy-duty mobile producer-gas plant, was sucked through. Charcoal

powder having particle size in the range of 1-1,000 microns was dusted at the inlet end of the filter unit for a period of ten minutes. The dust, which passed through the filter bed, was collected on the filter paper and examined under a microscope for pore size determination. In every case the predominant size of charcoal dust particles was determined. The total number of observations by various workers were 24 in each case. The mean of these values together with the probable error are summarized in Table II. These results have been obtained while using the linear filter.

A radial filter is shown diagrammatically in Fig. 2. It consists of a cylindrical shell of 10" diameter by 24" long. Inside it, there are two co-axial expanded metal cages 21½" long and 8½" and 3" outside and inside diameters respectively. The space between the two tubes was filled with the material under test. The packed unit was put inside the cylindrical shell. While packing the

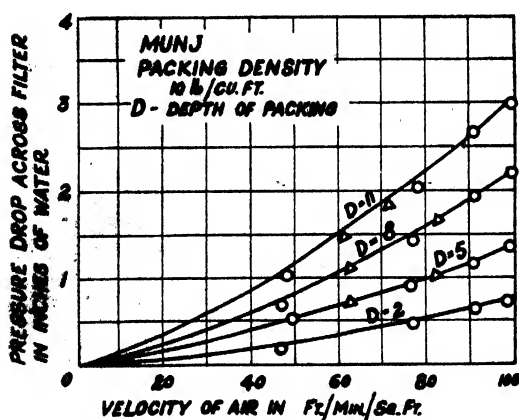
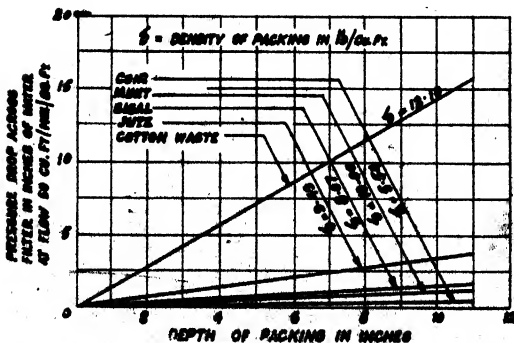
FIG. 7 — PRESSURE DROP VS. VELOCITY OF AIR FOR *munj*.

FIG. 8 — PRESSURE DROP VS. DEPTH OF PACKING FOR THE FIVE MATERIALS IN A LINEAR FILTER.

TABLE II

MATERIAL	FIBRE DIA., IN MICRONS	DENSITY OF PACKING, IN LB. PER CU. FT.	DEPTH OF PACKING, IN INCHES	SIZE OF PARTICLES COLLECTED ON FILTER PAPER, IN MICRONS
Cotton waste	428†	12.10	2	37.2 ± 2.2
			5	21.0 ± 1.9
			8	16.6 ± 1.9
			11	9.7 ± 1.9
Jute	73	9.49	2	*120 ± 7.2 (?)
			5	19 ± 1.9
			8	13 ± 1.9
			11	6.8 ± 1.9
Sisal	158	8.07	2	216 ± 7.2
			5	215 ± 7.7
			8	210 ± 8.2
			11	*67.4 ± 7.2
Coir	360	5.54	2	281 ± 13.9 (?)
			5	249 ± 7.2
			8	242 ± 11.1
			11	212 ± 7.2
Munj	228	10.00	2	465 ± 19.8
			5	445 ± 31.6
			8	459 ± 7.2
			11	339 ± 7.2

† In this case, dia. of yarn is given.

\* Irregular values.

material in the cages and fitting them inside the filter, every possible care was taken to avoid any short circuit paths for gas. In order to measure the pressure drop, a water manometer was connected across the filter bed. The results of pressure drop at various rates of air velocity at maximum packing density for the five materials have been graphically represented in Fig. 9. The pressure drop for the empty filter was also recorded.

### Discussion

A study of the pressure drop curves for the various materials show that cotton waste has exhibited the highest and *munj* the least resistance to the flow of gas. A glance at Table II reveals that the size of dust particles passing through the filter bed decreases with an increase in the depth of packing. The compressibility and the fibre diameter of the material, in general, play an important part in the dust-collecting efficiency.

From the characteristics of the materials and the size of dust particles passing through the filter bed (see TABLES I and II), the various materials investigated may be divided into three groups:

Group I — Cotton waste and jute.

Group II — Sisal and coir.

Group III — *Munj*.

Group I — Of all the materials investigated, cotton waste and jute have been found to

be the best. Cotton waste offers a high resistance to the gas flow. It cannot be easily cleaned and, therefore, the chances of its being used repeatedly for gas filtration are limited. But, in spite of these shortcomings, the dust-retention capacity of cotton waste is very prominent. During the various gas purity tests conducted in these laboratories, it has been most frequently noticed that the colour of the standard filter paper used to arrest the particles of dust present in the gas as finally supplied to the engine, does not appreciably change throughout the dust test when using cotton waste in one of the filters of the gas producer. From the summary of the results given in Table II one is led to think that jute is as good a filtering medium as cotton waste. As already stated, the criterion of a good filtering medium is that it should allow the least quantity of the smallest dust particles through the filter bed. Considering the gas filtration qualities of the two materials, cotton waste is found to be superior to jute and is most suitable for use in the second stage of producer-gas filtration. Though the size of dust particles passing through the jute filter bed is almost the same, yet it allows to pass slightly more quantity of dust and increases the work of the final filter. This could, however, be compensated by increasing the depth of packing of the filter bed. Jute is recommended in place of cotton waste for use in the second stage of gas

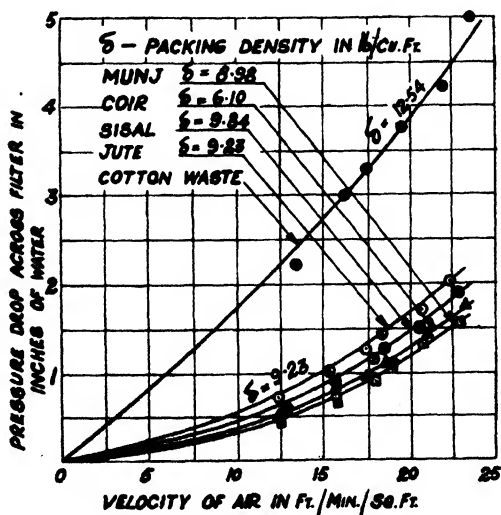


FIG. 9 — PRESSURE DROP VS. VELOCITY OF AIR FOR THE FIVE MATERIALS IN A RADIAL FILTER.

filtration, provided the depth of filter bed is increased.

*Group II* — Taking into consideration the size of particles passing through the filter bed, depth of packing, the quality of gas filtration, sisal has been found to be better than coir. Coir fibres are generally brittle and, therefore, the facilities of its repeated use for gas filtration are limited. There is, however, not much to choose between the two materials so far as the pressure drop through them is concerned. Of the two, sisal is to be preferred for efficient gas filtration, provided it is available in large quantities at a reasonable price. Coir and sisal are only suitable for use in the first stage of gas filtration.

*Group III* — Though *munj* has almost the same packing density as jute, yet the size of particles passing through its filter bed is the highest among the various filtering media studied in this investigation. Particles as big as 364 microns have been found to pass through the filter bed, even though the depth of packing and the packing density are maximum. This clearly points to its poor quality. The material is very difficult to pack and to spin. Such materials, therefore, are unsuitable for any stage of gas filtration.

By packing these five materials at maximum packing density in a radial filter, an idea of the resistance offered to gas flow has been obtained by the study of pressure drop

at various rates of air velocity. The results obtained in the two cases are quite consistent with each other.

In conclusion it may be stated that the data collected so far helps one in choosing the materials most suited for the first and second stages of gas filtration and in designing efficient radial filters so commonly used in India.

A mathematical study of the gas flow through these materials is in course of investigation.

The authors take this opportunity of thanking Dr. L. C. Verman, Director, Indian Standards Institution, New Delhi and Mr. K. A. Nair for the kind help and encouragement received from them during the course of the investigation.

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# Effect of the Water-soluble Matter in Jute on the Microbiological Deterioration of the Fibre

S. N. BASU

Indian Jute Mills Association Research Institute, Calcutta

LITTLE is known of the precise nature of the water-soluble matter in natural vegetable fibres. In cotton, the presence of 0.5 per cent malic acid and 0.07 per cent citric acid has been reported<sup>1</sup>. Some of the nitrogenous compounds present are probably water soluble. According to Marsh and Wood<sup>2</sup> raw cotton contains 0.2 per cent nitrogen. Jute has a nitrogen content of 0.2-0.3 per cent, nearly half of which is extractable by boiling water<sup>3</sup>. While these components and also soluble carbon sources may help fungal growth, they are present in the fibre in amounts probably too low to have any appreciable effect. On the other hand, substances of vitamin nature are required by living organisms only in comparatively minute quantities and may cause a striking growth response. The presence of such water-soluble growth factors or nutrilites in natural vegetable fibres is only to be expected and have actually been demonstrated, directly or indirectly, in some instances. Thus Robbins and Ma<sup>4</sup> showed that *Ceratostomella* species having deficiencies of thiamin, biotin and pyridoxine would grow normally when an extract of cotton batting was added to the medium. Robbins and Schmitt<sup>5</sup> found evidence for the presence, in cotton, of an unknown factor that would stimulate the germination of *Phycomyces* spores. Sherwood and Singer<sup>6</sup> demonstrated the presence of folic acid in cotton bolls and even in absorbent and non-absorbent cotton linters. Constantin<sup>7</sup> reported in carded cotton a water-soluble substance promoting the growth of yeast which did not appear to be any of the known members of the vitamin B complex. Buston and Basu<sup>8</sup> have established the presence of nicotinic acid, thiamin, riboflavin, pyridoxine, pantothenic acid and biotin in the water extract of jute; quantitative assay showed that the raw fibre contained 0.116-0.471  $\mu\text{g}$ . of these vitamins per gm. except for biotin; this substance, active in much smaller amounts, was present in a lower concentration than the others.

Many known vitamins — and probably some yet unidentified — play a part in the metabolism of fungi as in many other living organisms, but while some fungi can synthesize their own requirements of all of these compounds, others cannot. These latter would not grow well on synthetic media but would do so on natural materials due to the presence there of these growth factors. That vegetable fibres, and fabrics made therefrom, should support good growth of such deficient organisms is, therefore, not surprising. This phenomenon is not of much direct economic importance in the textile industry in the case of many so-called superficial fungi but its industrial significance cannot be overlooked when active cellulose decomposing species are found to suffer from vitamin deficiencies.

Cases of such fungi responding to the stimulation of micro-nutrients have been reported in recent years. Zuck and Diehl<sup>9</sup> have found that certain slow-growing species, not commonly associated with cellulose decomposition, nevertheless cause loss in strength of cotton duck; these were able to grow and sporulate well on cotton extract agar but not on synthetic sugar-based media. Zuck<sup>10</sup> also found cotton extract agar a particularly good medium for growing the closely related genera *Memnoniella* and *Stachybotrys*; these organisms, very well known as cellulose decomposers and believed to cause extensive damage to cotton stores, have later been found to require pre-formed biotin for growth<sup>8,11</sup>. It was shown by Buston and Basu<sup>8</sup> that jute extract had the same effect on *Memnoniella* as the corresponding amount of pure biotin. The growth and perithecial fruiting of another well-known cotton-destroying species *Chaetomium globosum* was also shown by the same authors to be markedly stimulated, but this effect was believed to be due to an yet-unidentified factor in the jute extract.

In view of these previous findings it was considered worth while to investigate, in terms of actual loss in strength, whether the



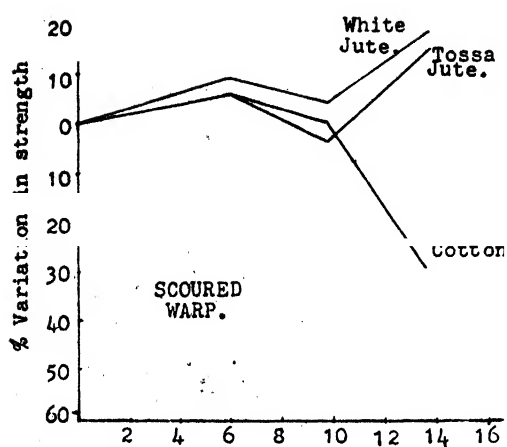


FIG. 1 — WEEKS OF INCUBATION.

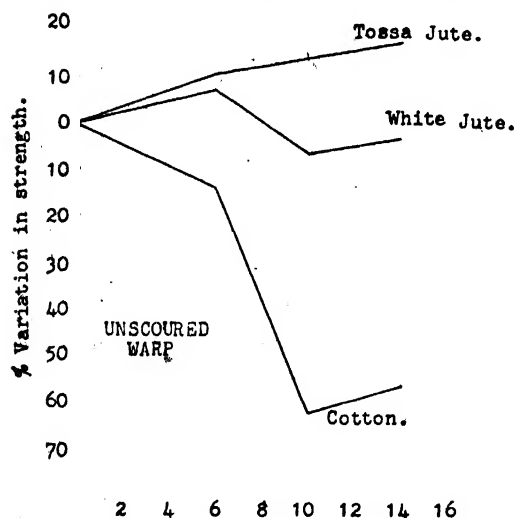


FIG. 2 — WEEKS OF INCUBATION.

water-soluble fraction of jute fibre did actually influence its deterioration and if so to what extent. The results presented below substantiate the expectation that the effect of the water-soluble constituents on the biological decay of the fibre is by no means negligible.

#### Experimental

In the first instance, the rates of deterioration of scoured and unscoured canvases made from white jute (*Corchorus capsularis*) and tossa jute (*C. olitorius*) and also from cotton were compared. Scouring was done by steeping in a solution of 1 per cent soap and 0.5 per cent soda ash for 1 hr. at 80°C.

The samples were rinsed in water and, after inoculation with soil extract, incubated in a humidity box highly charged with spores of numerous mildew fungi and containing water at the bottom. Portions were withdrawn at intervals and loss in tensile strength determined in the usual way. The results shown graphically in Figs. 1-4 demonstrate that scouring improved mildew resistance in all cases (see also Figs. 5-8). Incidentally, it may also be noted that cotton appears to be more susceptible to rot than jute, and that there was no definite difference between tossa and white jutes so far as this property was concerned.

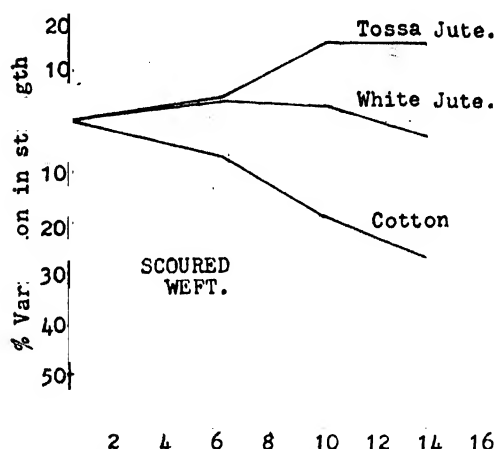


FIG. 3 — WEEKS OF INCUBATION.

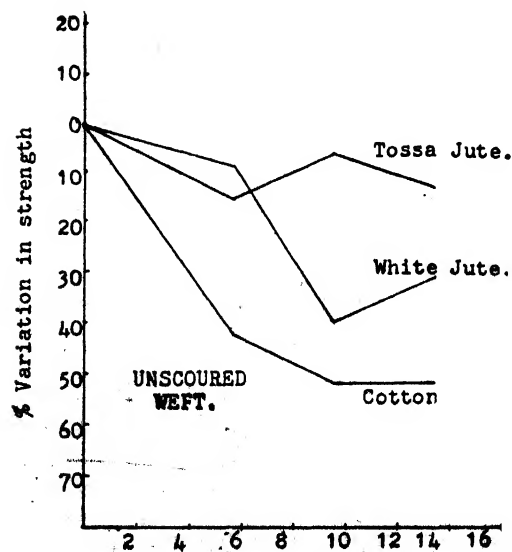


FIG. 4 — WEEKS OF INCUBATION.

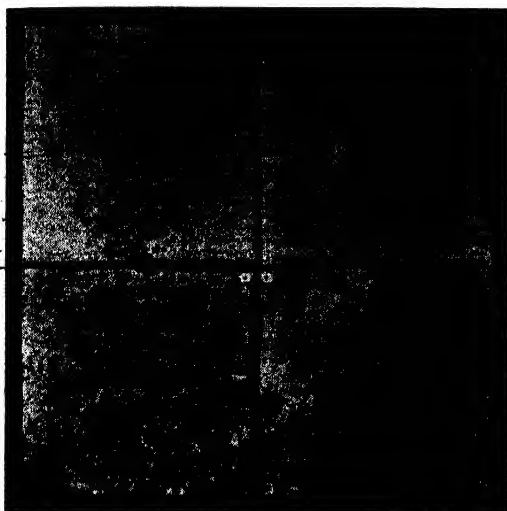


FIG. 5 — UNSOURED JUTE ( TOSSA ).

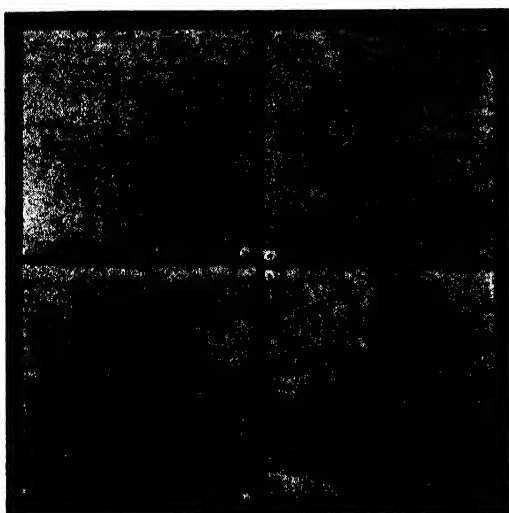


FIG. 6 — SCOURED JUTE ( TOSSA ).

While scouring much improved mildew resistance, this treatment probably removed somewhat more matter than the water-soluble fraction. In the next experiment samples of hessian cloth and jute fibre were leached in boiling distilled water for 1 hr. After air drying, these were inoculated by pressing against a heavily mildewed cloth. The samples along with the untreated controls were incubated over water in a desiccator. The day temperature varied from 23°-38°C. Microscopic examinations were made at monthly intervals with the following results ( TABLE I ).

At the end of 3 months, the fibre samples were subjected to the microscopic test devised by Macmillan and Basu for the detection of damage on jute<sup>12</sup>. Briefly, the test consists in swelling a number of fibres according to a particular procedure when damaged and undamaged fibres appear different. The percentage of damaged fibres is expressed as "damage count". This factor is a measure of the tensile strength inasmuch as it has been found to increase with decreasing strength of the sample. Weighed portions of the test samples and of a normal

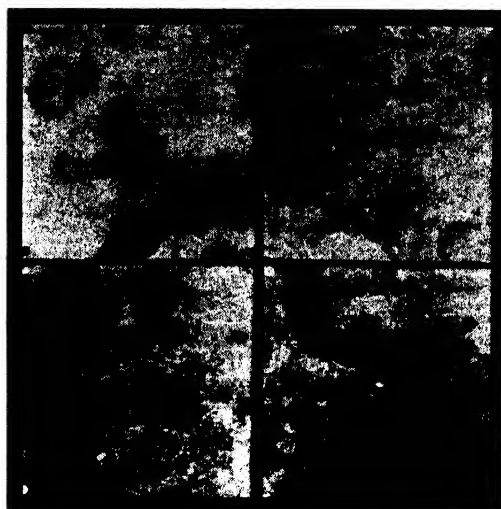


FIG. 7 — UNSOURED COTTON.

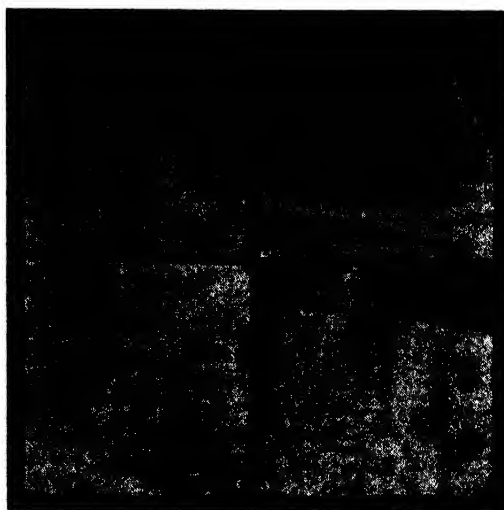


FIG. 8 — SCOURED COTTON. (THE STRAIGHT-EDGED PATCHES ARE NOT DUE TO MILDEW.)

TABLE I—FUNGAL GROWTH ON LEACHED &amp; UNLEACHED MATERIALS

INCUBATION PERIOD	UNLEACHED		LEACHED	
	Fabric	Fibre	Fabric	Fibre
1 month	Moderate growth	No detectable growth	Mild growth	No detectable growth
2 months	Moderate growth; visible peritheca of <i>Ch. indicum</i>	Visible peritheca of <i>Ch. indicum</i>	Mild growth	No detectable growth
3 months	Heavy growth; numerous peritheca of <i>Ch. indicum</i>	Moderate growth; many peritheca of <i>Ch. indicum</i>	Mild growth; no <i>Ch. indicum</i>	No detectable growth; no <i>Ch. indicum</i>

fibre were also plated out on a suitable medium to determine the nature and extent of growth. The results are set out in Table II.

Here also it is seen that attack on the unleached sample proceeds much more rapidly than on the leached fibre. It is interesting to note that *Chaetomium indicum* which actively decomposes jute and cellulose<sup>3</sup> and which seems to be a native infection on jute frequently developing on the rotting fibre<sup>1a</sup> showed profuse growth on the unleached fibre but no growth on the leached sample. The *Phycomycetes*, which are known to be devoid of any cellulose decomposing power, were absent on the leached fibre which suggests that these species thrive on the superficial nutrients.

The stimulating action of the water extract of jute was then more directly demonstrated by comparing the growth rates of species on a synthetic medium with and without added jute extract. Liquid Czapek-Dox medium (neutral) with 1 per cent glucose was the basal medium and growth on this was compared with that on the same medium plus an extract of 2 gm. of jute for every 100 c.c. In Figs. 9-10, the positive acceleration of growth by jute extract can be seen. Of the species tested, *Chaetomium globosum* and *Memnoniella echinata* are well-known cellulose decomposers, although rarely found on jute materials. That substances of the nature of growth factors stimulate the growth and sporulation of these species is already known<sup>9-11</sup>. On the other hand, *Chaetomium indicum* and *Aspergillus terreus* grow and sporulate readily on ordinary synthetic media. It is interesting to note that these species

TABLE II—DAMAGE CAUSED &amp; NATURE OF ATTACK ON LEACHED &amp; UNLEACHED SAMPLES

TEST	UNINCUBATED FIBRE Normal	INCUBATED FIBRE	
		Leached	Unleached
Damage count	12-14	22	32
Fungal count (per gm.)	30,000	60,000	200,000
Fungal sp. isolated	<i>Pen. citrinum</i> , <i>Asp. terreus</i>	<i>Pen. citrinum</i> , <i>Asp. terreus</i> , <i>Asp. glaucus</i> , <i>Paecilomyces varioti</i>	<i>Pen. citrinum</i> , <i>Asp. terreus</i> , <i>Ch. indicum</i> , <i>Phycomycetes</i>

which are frequently isolated from rotting jute materials are also markedly stimulated by jute extract (see FIG. 10).

### Discussion

It is unlikely that the effect observed was due to the presence in the jute extract of nutrients such as particularly readily available sources of carbon or nitrogen. The extract was found not to contain any sugar, and the nitrogenous matter in it, although qualitatively an excellent nutrient for many species<sup>3</sup>, must have been quantitatively inadequate. In the last experiment reported above, calculation shows that jute

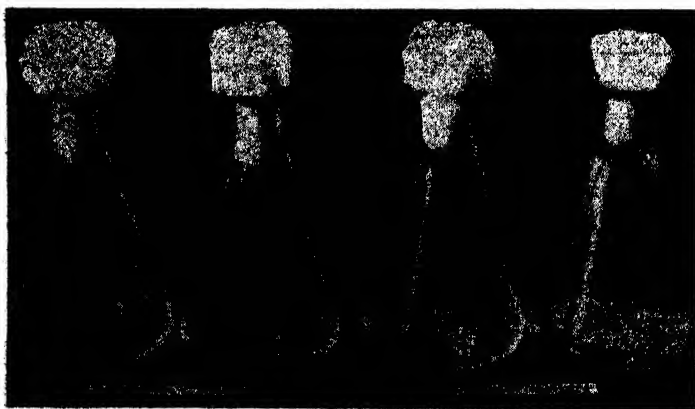


FIG. 9—GROWTH OF *Memnoniella echinata* (AT 22°C.) AND *Chaetomium globosum* (AT 30°C.) AFTER 6 DAYS, WITHOUT AND WITH JUTE EXTRACT.

extract supplied only some 6 per cent of the total nitrogen present in the medium. Moreover, it had been found previously in the case of *Chaetomium globosum* that organic nitrogen sources did not produce much better growth than nitrate<sup>8</sup>.

Fungi are also known to be markedly stimulated by traces of mineral elements<sup>14</sup>. Beeson<sup>15</sup> has shown the presence of K, Ca, Mg, P and S in raw cotton. According to Marsh and Wood<sup>2</sup>, raw cotton gives 1 per cent ash which has in it Ca, K, Mg, Al and Fe. Parsons<sup>16</sup> reports 0.8-1.5 per cent ash in jute and he quotes Cross and Bevan as showing it to consist chiefly of Si, K, P with traces of Mn and Cu. Ca and Fe are also probably present. It is possible, therefore, that the stimulatory effect of jute extract is due to the oligodynamic action of mineral micro-nutrients. However, these elements are required in such minute traces that enough of them are probably present in synthetic media prepared from chemicals not specially purified, so that it would be difficult to explain the results shown in Figs. 9 and 10. That stimulation was caused by growth substances of vitamin nature remains, therefore, the most likely possibility, particularly in view of previous evidence as summarized in the introduction.

From the results obtained it would seem likely that the simple measure of hot water leaching, if applied on the finished goods or the raw jute fibre, should appreciably reduce mildew trouble in actual use of jute materials.

### Summary

The water-soluble fraction of jute fibre has stimulatory action on many fungi including those known to attack strongly jute and cellulosic materials. This is probably due to the presence in it of growth factors of vitamin nature. Fibre and fabrics boiled in water actually showed increased mildew resistance.

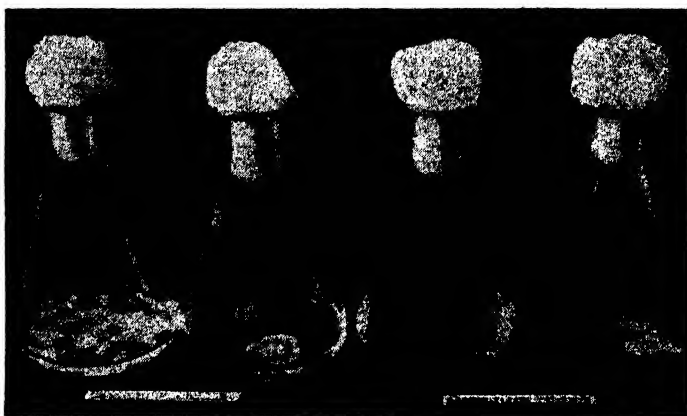


FIG. 10 GROWTH OF *Chaetomium indicum* AND *Aspergillus terreus* AFTER 5 DAYS (AT 30° C.), WITH AND WITHOUT JUTE EXTRACT.

### Acknowledgements

The author wishes to thank Mr. S. N. Ghose, M.Sc., for his help with some of the tests, and the Committee of the *Indian Jute Mills Association Research Institute* for permission to publish the results described.

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# Water Content of Lime-puzzolana Mortars

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**A**DDITION of correct amount of water in lime-surkhi mortar has a direct bearing on its strength development. Studies carried out at the *Building Research Station, D.S.I.R., England*<sup>1</sup>, show that 2 : 1 : 9 puzzolana : hydrated lime : standard sand mortar gives higher strength at 12 per cent moisture as compared with 14 and 16 per cent. Still further increase is noticed at 8 to 10 per cent moisture.

It has been shown by Khan and Verman<sup>2</sup> that dry mixes are superior to wet mixes. In this investigation two discrete values of consistency were used to determine the effect of consistency on the strength development of lime-surkhi mixes. The results obtained in these experiments indicated the superiority of only one value over the other. It was further pointed out by them that there seems to be one particular consistency at which the strength of lime-surkhi mortar is maximum. In the present investigation the scope of work was further extended and the strength of lime-surkhi mortar was determined at different water contents. The ratio of lime to surkhi was also one of the variables.

## Experimental

Four samples of bricks were collected for the purpose of the present investigation : two from a brick-field, Roorkee, and two from a brick-field, Ballawali, a place twenty miles east of Roorkee. By visual examination the bricks were grouped into well-burnt and under-burnt bricks. It may be pointed out that bricks collected from these fields do not represent burnt clay alone but a mixture of sand and clay in unknown proportions. No attempt was made to ascertain the exact proportion of sand and clay mixture used in the manufacture of bricks, as the composition of soil changes from one brick-field to the other and in the same brick-field. Both types of bricks were powdered by means of a wooden hammer until the surkhi so obtained could be passed through a 30-mesh sieve.

The composition of the slaked lime used in the experiments is given in Table I. Before using it for the purpose of making test briquettes, it was also passed through a 30-mesh sieve.

TABLE I — CHEMICAL COMPOSITION OF LIME

	%
Silica-SiO <sub>2</sub>	1.84
Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub>	1.64
MgO	6.01
CaO	65.76
Ignition loss	24.87
Sulphuric anhydride	0.10
Alkaline salts as NaCl and KCl	0.20

The test briquettes were matured for 28 days. The initial maturing for 7 days was done in a damp box at a relative humidity of 90 to 95 per cent. For the rest of the period maturing was done under tap water containing 18 parts of soluble salts in 100,000 parts of water. The briquettes were matured at room temperature. The maximum and minimum temperatures were recorded during the day. The mean of average daily temperatures during the period of maturing are shown in Table II.

Batches of briquettes after maturing period were tested for their tensile strength. Each batch consisted of 6 briquettes. The average of test results was taken to be the tensile strength of the batch. Further the briquettes were gauged by the same operator under uniform conditions.

Further, 200 gm. of lime-surkhi mix was subjected to compaction in a compacting cylinder having a diameter of 2.5". It was compacted in one layer receiving 15 blows from a 6.6 lb. hammer falling through a height of 10".

## Discussion

The variation of strength due to changes in the moisture content of lime-surkhi mortar under the test conditions is clearly demonstrated. It is seen that maximum strength at 1:7 and 1:4 lime-surkhi ratios is obtained between 25 to 28 per cent moisture

content, while it requires a higher water content to get the maximum strength at 1:1 lime-surkhi ratio.

It has been observed that when lime-surkhi mix is compacted, cracks are developed at a particular moisture content. The result of compaction experiments on surkhi sample B are given in Table III. It is to be noted from the results obtained that at 1:7 lime-surkhi ratio cracks are produced at 27.08 per cent water content, while at 1:4 and 1:1 lime-surkhi ratios the development of cracks occurs at 27.50 per cent and 33.90 per cent respectively. It may thus be concluded from the results shown in Table II and Table III that maximum tensile strength of lime-surkhi mix is obtained just below the moisture content at which cracks develop. The strength of the mix decreases as the

percentage of moisture in the mortar is raised above this limit.

It may be further pointed out that the minimum tensile strength of lime-surkhi mortar is obtained in the proportion of 1:1 as compared to the ratios 1:7 and 1:4 except in surkhi C where the strength is low on the whole. However, it is difficult to compare the strength of the mortars at 1:7 and 1:4 ratios as the difference in strengths shows such variations that it is easily masked by the effect of change in temperature.

Work carried out in *Building Research Station* in United Kingdom<sup>3</sup> has shown that the temperature coefficient of the rate of strength development of lime-puzzolana-sand mixes is very high. It is also clear from the results of the present investigation that during maturing temperature has a

TABLE II — TENSILE STRENGTH OF LIME-SURKHI MORTAR AT DIFFERENT WATER CONTENTS

SAMPLE NO.	RATIO OF LIME TO SURKHI								
	1:7			1:4			1:1		
	MOISTURE %	TENSILE STRENGTH IN LB. PER SQ. IN.	TEMP. °F.	MOISTURE %	TENSILE STRENGTH IN LB. PER SQ. IN.	TEMP. °F.	MOISTURE %	TENSILE STRENGTH IN LB. PER SQ. IN.	TEMP. °F.
A Well-burnt surkhi from Roorkee	22.9	46	74.3	22.8	45	72.5	27.6	10	69.8
	26.1	90		26.0	83		30.3	30	
	28.9	70		29.1	76		33.0	47	
	32.0	40		32.1	51		37.9	30	
	34.8	37		35.0	31		42.8	27	
B Under-burnt surkhi from Roorkee	23.6	103	83.4	23.0	54	88.5	29.0	100	90.2
	27.3	170		27.3	177		32.4	105	
	30.1	140		30.0	167		35.3	79	
	33.0	121		33.1	153		37.3	74	
	35.5	106		35.5	135		39.1	64	
C Well-burnt surkhi from Ballawali	23.2	10	60.3	24.3	8	57.0	27.5	9	56.2
	25.0	11		27.5	12		30.6	14	
	27.2	11		30.4	12		33.4	12	
	30.4	10		33.5	6		36.5	11	
	33.4	7		35.1	5		39.5	10	
D Under-burnt surkhi from Ballawali	25.0*	102	100			69.9			73.3
	23.6	91		23.5	102		29.8	72	
	26.4	98		26.4	108		32.5	81	
	29.5	83		29.5	88		35.4	58	
	32.6	72		32.4	72		38.6	50	
	35.4	52		35.5	57		41.3	48	
	26.4*	153	100				44.5	41	

\* represents tensile strength after 14 days maturing.

TABLE III — FORMATION OF CRACKS IN SURKHI SAMPLE B

No.	RATIO OF LIME TO SURKHI					
	1:7		1:4		1:1	
	MOISTURE %	FORMATION OF CRACKS	MOISTURE %	FORMATION OF CRACKS	MOISTURE %	FORMATION OF CRACKS
1	23.45	No cracks	23.70	No cracks	31.90	No cracks
2	24.96	do	25.44	do	33.50	do
3	25.50	do	26.37	do	33.80	Fine cracks
4	26.68	do	27.21	do	34.01	Cracks
5	26.96	do	27.50	Fine cracks	34.73	do
6	27.08	Fine cracks	27.67	Cracks	35.50	do
7	27.18	Cracks	28.55	do	36.30	do
8	27.52	do	29.95	do	...	...

marked effect on the strength development of lime-surkhi mixes; the higher the temperature, the greater is the strength developed in a given time. Comparing the results of well-burnt surkhi C and under-burnt surkhi B we find vast differences in strength. This much of difference cannot be explained due to a difference in the chemical composition of surkhi. The effect of temperature is also to be considered. In order to confirm this point further samples of surkhi C and D from Ballawali were matured for a period of 14 days at 100°F. It is clear from the results obtained that after 14 days, tensile strengths of the two samples matured at 100°F. are much higher than the same samples matured at room temperatures for 28 days (TABLE II). So it leads us to the fact that in all studies on lime-surkhi mortars the effect of temperature on its strength development is of fundamental importance. It may be mentioned here that Singh and Bahri<sup>4</sup> did some work on the incorporation of puzzolana in kanker lime. Better results could have been obtained if the workers had taken into consideration the effect of temperature also. Unless a systematic study of the subject is made, it may be too early to suggest that when early strength development in a mortar is desired, the construction with lime-surkhi mortar should preferably be undertaken in the summer months of the year.

Investigations carried out at *Building Research Station* in U.K.<sup>5</sup> have established that for every clay there exists a temperature of firing (between 600°-800°C.) which gives it the highest activity. Heating at a temperature higher than 1,000°C. appears to destroy the puzzolanic activity of clay. Studies of Khan and Verman<sup>3</sup> conclusively proved that higher temperature of burning gave higher strength of surkhi when used in lime mortar. However, in the above investigations there are samples of under-burnt surkhi which show, at initial stages of maturing, superiority over well-burnt surkhi. In the present investigation also under-burnt surkhi seems to give better early strength as compared with well-burnt surkhi. Probably the chemical composition of clays plays an important part in determining the optimum temperature at which a puzzolana of highest activity is obtained. It may be mentioned here that we have 2 major soil groups in this country: brown to black soils of the Deccan Trap and the Indo-Gangetic alluvium. Besides, there are red soils of Madras and lateritic soils of Mysore.

These soils differ widely in their physical properties and chemical composition. It may be pointed out that results obtained by Khan and Verman are based only on surkhi made from Bengal soils. Though the clays used in the experiments were of varied nature, still it might be interesting to extend the scope of the work to other types of soils available in different parts of the country.

In practice one finds that wet consistency is often employed. This practice has proved to be harmful from tensile strength point of view. Moreover, as shown above, when lime-surkhi mortars are subjected to compression at wet consistency, cracks are developed. This provides a greater scope for the penetration of rain in the structure.

It may be pointed out here that mortars which exhibit high tensile strength are not necessarily those which adhere most strongly to all types of bricks. A high tensile strength is valueless unless there is a good adhesion to the brick on each side. Thus, actual recommendations as to the quantity of the water to be used in lime-puzzolana mortar in actual construction cannot be made unless a study of the effect of moisture on the adhesive strength of the mortars is also made. Proper allowance will have to be given for the absorption of water by the brick from the mortar.

### Conclusions

There is a critical moisture content value at which the early strength of lime-surkhi mortar is maximum. Further, when lime-surkhi mix is subjected to compaction at higher moisture content, cracks are produced.

As the percentage of lime increases in the mortars, the quantity of water required to get the maximum strength also increases.

The early strength of lime-surkhi mix at 1:1 ratio is the minimum as compared to other ratios, except in surkhi C.

The effect of temperature on the strength development of mortar is very pronounced. Higher rate of strength development is obtained at higher temperature.

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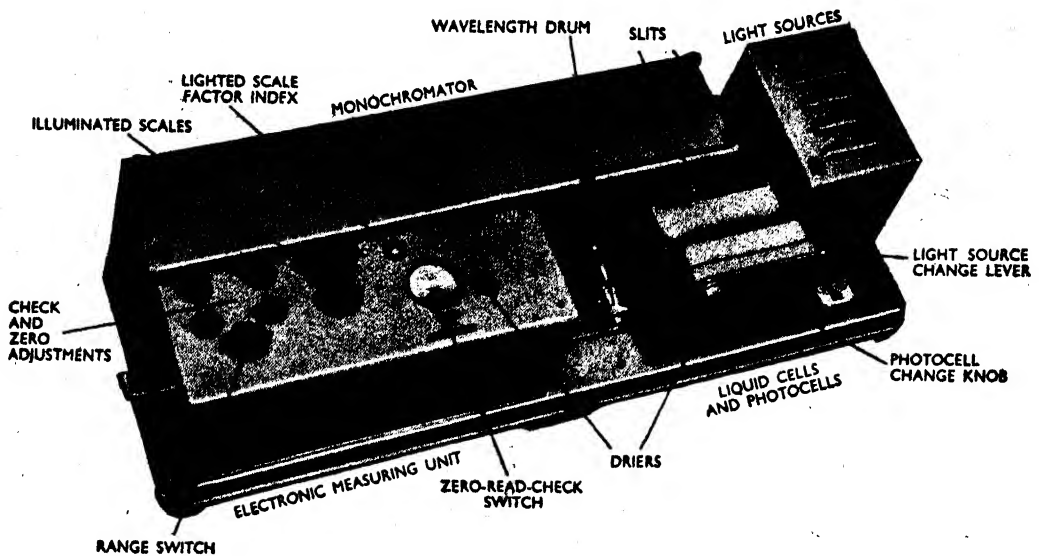
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# Hypochlorite Oxidation of Cotton Cellulose in Presence of Mixtures of Certain Vat Dyes — Part I

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THE ability of certain vat dyes to accelerate the oxidation of cellulose by air and by chemical oxidizing agents in presence of light is well established<sup>1,2</sup>. It has been shown that nearly all yellow, orange and several red, pink and brown vat dyes behave in this manner. None of the blue and green vat dyes exhibit this behaviour. In the dark, however, the oxidation of cellulose by the oxidizing agent has been found to be independent of the colour of the dye present on the fibre. This behaviour of vat dyes was explained by Scholefield and Turner<sup>3</sup> on the basis of a chemical theory, which may be summarized as follows: Firstly, illumination is necessary for the acceleration of the oxidation of the cellulose substrate and active dyes are more or less restricted to certain colour groups only. Secondly, the active yellow and orange vat dyes show a strong absorption of light of short wave length — between 3,600 to 4,000Å — while the non-active blues and greens do not show strong absorption in this region<sup>4</sup>. Scholefield and Turner<sup>3</sup> assumed that the yellow and orange vat dyes, by virtue of their ability to absorb light in the near ultra-violet region, where large light quanta are available, bring about the necessary chemical change. They further assumed that the initial chemical change brought about by the absorbed light is to reduce the vat dyestuff to its leuco compound and at the same time to cause an equivalent oxidation of the cellulose substrate. The leuco vat dyestuff thus formed then gets oxidized by air according to circumstances, also bringing about the

auto-oxidation of cellulose, as suggested by Engler<sup>5</sup>. Scholefield and Turner<sup>3</sup>, therefore, deduced that the chemical production of leuco compounds of vat dyes dyed upon the fibre should be capable of accelerating the oxidation of cellulose by oxidizing agents in the absence of light and irrespective of the optical properties of the vat dye.

This point was examined in considerable detail by Turner, Nabar and Scholefield<sup>6,7</sup>, who showed that when a number of tendering and non-tendering leuco vat dyes on cellulose were treated with dilute hypochlorite solutions in the dark, a considerable amount of the acceleration of the hypochlorite action on cellulose is brought about. The extent of the modification depended on the nature of the vat dyestuff present on the cellulose. With dyes which themselves underwent modification by the hypochlorite treatment, the extent of modification in the cellulose substrate was lower than that when the dyes were unaffected by hypochlorite. The acceleration of cellulose oxidation during the air oxidation of leuco compounds of vat dyes chemically produced in the dark has also been demonstrated by Breare and Turner<sup>8</sup>.

Another interesting aspect of the behaviour of tendering action of active vat dyes is their behaviour in the presence of a non-active blue or green vat dye on cellulose. Scholefield and Patel<sup>9</sup> investigated the action of light on dyeings prepared from mixtures of active and non-active vat dyes. They found that in such mixtures the light-resistant blue was rapidly destroyed when an active yellow or an orange vat dye was

present. They further showed that when mixtures of vat blues and active yellows were exposed in their leuco state to light and air, the blue is in certain cases wholly or partially destroyed. Ciba Blue 2B was found to be especially susceptible in this respect. The same behaviour was observed when the mixed dyeings were subjected to the action of chemical oxidizing agents in the presence of light. Similar observations have been recorded by Scholefield and Stafford<sup>10</sup>, König<sup>11</sup> and Jones<sup>12</sup>.

The present investigation relates to the behaviour of a mixture of a non-tendering dyestuff with an active orange or yellow towards the action of dilute sodium hypochlorite solution. From among the various tendering and non-tendering vat dyes, it was decided to select Ciba Blue 2B to represent the non-tendering class and Cibacron Orange R to represent the tendering class. Dyeings on cotton were prepared using various concentrations of the two dyestuffs individually and in mixture on cotton yarn. These dyeings, both in the reduced and unreduced state, were then subjected to the action of hypochlorite solution under controlled conditions of concentration of available chlorine, pH and temperature for 10 min. The available oxygen lost from the hypochlorite solution as a result of the treatment was estimated and the treated dyeings after thorough washing, drying and conditioning were examined for their cuprammonium fluidity and copper number. The degree of fading or destruction of the blue component was visually noted.

## Experimental

Sodium hypochlorite used in the investigation was prepared according to the methods of Musprat and Smith<sup>18</sup>. The stock solution had the following characteristics: free alkali, 2.0-2.5 N; active chlorine, 150-160 gm./litre; chlorate and carbonate, nil. For the oxidation experiments the dilute hypochlorite solutions were buffered to pH 9.1 using M/20 borax solution<sup>19</sup>.

**Available Chlorine**—The concentration of active chlorine in buffered sodium hypochlorite solutions used for oxidation was adjusted, as near as possible, to 3 gm. available chlorine per litre. The pH of the solutions was determined by using the Morton type glass electrode system and a valve potentiometer<sup>20</sup>.

**Cotton**—Fully bleached 18's yarn manufactured from Indian cotton was used for

oxygen consumption experiments. The grey yarn was subjected to standard bleaching treatment ensuring more or less complete removal of impurities from the cellulose. After thorough washing, drying and conditioning, the cotton had the following properties: cuprammonium fluidity<sup>13</sup> (0.5 per cent solution), 7.8; copper number<sup>14</sup> (Heyes' method), 0.15; milliequivalents of  $-\text{COOH}/100$  gm. of cellulose, 0.5.

**Method of Dyeing**—The dyestuff was pasted with a small quantity of monopotassium soap and dispersed in 2 litres of cold water. Calculated quantities of caustic soda and hydrosulphite were added while stirring the mixture. It was then gradually heated to 60°C. and then allowed to vat at this temperature for 30 min. 100 gm. of cotton yarn was wetted out with water containing a little alkali and hydrosulphite. It was squeezed and then worked in the dye solution for 45 min. The cotton, as far as possible, was kept immersed below the surface of the liquor during the dyeing. It was then removed from the dye-bath and excess liquor evenly wrung out. The dyeing was then kept immersed in cold running water till the oxidation of the dye was complete. This took about 2 hr. It was squeezed, allowed to dry, conditioned in the dark and stored away from dust and light.

**Reduction of the Dyeing**—10 gm. of the dyed yarn was treated with cold 2 per cent sodium hydrosulphite solution for 3 min. (The reduction of the dyestuff takes place within the first half minute after treatment with hydrosulphite solution.) The dyeing was then removed from the hydrosulphite bath, excess solution wrung out rapidly and the reduced dyeing immersed at once in cold distilled water from which air had been expelled by previous boiling. It was kept stirred below the surface of water for 1 min. and then transferred to another similar bath. This was repeated 5 times. Finally, the yarn was squeezed between fingers to remove the excess water and at once transferred to a hypochlorite solution suitably buffered and kept ready in a stoppered flask. The repeated washings of the reduced dyeing were sufficient to remove the impurities carried from the reduction bath by the cotton.

**Hypochlorite Treatment of Reduced Dyeings & Determination of Oxygen Consumed**—The method is similar to the one used by Nabar, Scholefield and Turner<sup>7</sup>. 10 gm. of the reduced vat dyeing were used for oxidation

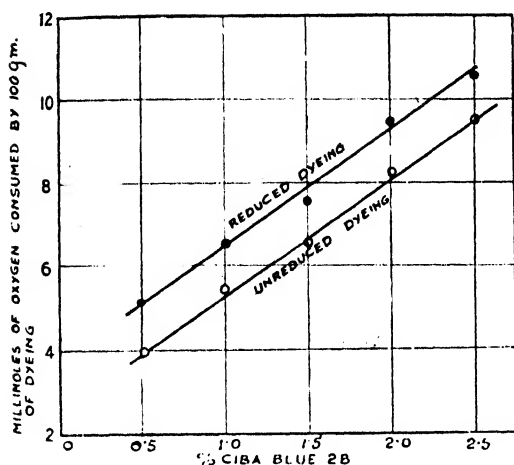


FIG. 1 — RELATION BETWEEN PERCENTAGE OF CIBA BLUE 2B USED FOR THE DYEING AND THE AMOUNT OF OXYGEN.

experiments. It was found that this weight of cotton approximately displaced 10 c.c. of water. It is impossible to get the reduced and washed dyeings in the dry condition and it is necessary to transfer the material to the hypochlorite solution in a wet state. The cotton, therefore, carries with it an undetermined weight of water and dilutes the hypochlorite. To allow for this dilution, the following procedure was adopted.

A flat-bottomed flask was graduated to hold 560 c.c. (550 c.c. for the liquor and 10 c.c. for cotton). Into this flask were introduced 500 c.c. of buffered sodium hypochlorite solution and the wet dyeing. The volume was then made up to 560 c.c. with cold distilled water. This diluted the original 500 c.c. of the hypochlorite solution to 550 c.c. A control experiment in which 227.3 c.c. of the stock solution was diluted to 250 c.c. with distilled water was carried out. After the hypochlorite treatment (10 min.), the liquor which remained was analysed for available chlorine and its final pH. From the difference in the concentrations of available chlorine in the control experiment and the solution after treatment, the net amount of oxygen used during the treatment can be calculated. The pH of the solution remained unaffected.

The reduction and oxidation of the dyeing was carried out in a dimly lit room. The hypochlorite solution used was standardized to contain 3 gm. available chlorine per litre. The time of treatment was 10 min. During the treatment, the dyeing was stirred

below the surface of the liquor to ensure uniform contact.

The oxidized cotton was washed free of chlorine.

**Cuprammonium Fluidity** — The cuprammonium fluidity of the various oxidized samples was determined by the standard B.C.I.R.A. method<sup>13</sup>.

**Copper Number** — The copper numbers were determined by the Schwalbe-Braidy method developed by the B.C.I.R.A.<sup>16</sup> and adapted for use with smaller quantities of the sample by Heyes<sup>14</sup>.

### Experimental Results

A synopsis of the experimental results is presented graphically in Figs. 1-8.

In this series of experiments, the concentration of Ciba Blue 2B and of Cibacron Orange R was varied and the dyeings were treated in the reduced as well as in the unreduced state. The concentration of the dye, the oxygen consumption and the cuprammonium fluidity of the dyeing are given in Figs. 1-4. Corresponding values for the mixed dyeings are given in Figs. 5-8.

### Discussion

In discussing the experimental results, consideration has to be given to: (a) the effect of a non-active dye such as Ciba Blue 2B on the oxidation of cellulose when treated with hypochlorite solutions; and (b) its influence on the properties of a tendering dye such as Cibacron Orange R in bringing about the acceleration of the oxidation of cellulose.

A reference to results set out in Fig. 1 shows that Ciba Blue 2B dyed cotton, both in the reduced and unreduced state, when treated with sodium hypochlorite solution for 10 min., brings about a considerable

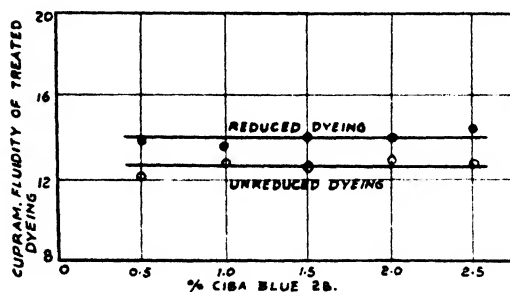


FIG. 2 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY OF THE DYEING AND THE CONCENTRATION OF CIBA BLUE 2B.

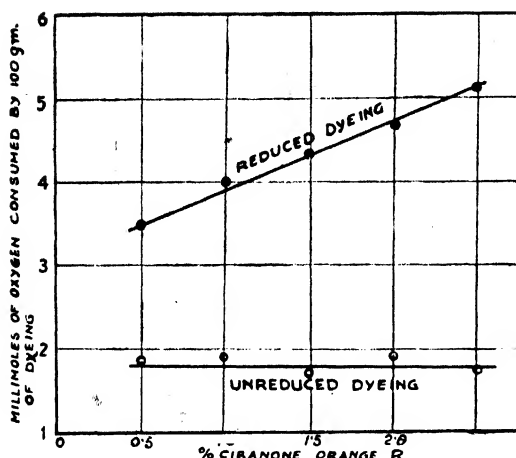


FIG. 3 — RELATION BETWEEN PERCENTAGE OF CIBANONE ORANGE R AND THE AMOUNT OF OXYGEN CONSUMED AND UNREDUCED DYEING.

amount of loss of active oxygen from solution. This amount of oxygen may be partly utilized in oxidizing the cellulose and partly in destroying the dyestuff. Close examination and comparison of the dyeings treated with hypochlorite solution both in the reduced and unreduced state with the untreated dyeings shows considerable loss of colour as a result of oxidation; so, at least part of the oxygen lost from the solution must have been used up for the destruction of Ciba Blue 2B. It is known that when Ciba Blue 2B dyed cotton is exposed to light and air for sufficiently long periods, the main product of oxidation of Ciba Blue 2B is di-bromo-isatin<sup>17</sup>. From Fig. 2 it is seen that the amount of degradation produced in the cellulose substrate is small. For an oxygen up-take of 0.32 per cent, a cuprammonium fluidity (0.5 per cent solution) of the order of 60 reciprocal poises may be expected, if all the oxygen is utilized for the oxidation of the substrate. It is found that the fluidity of the treated dyeings is of the order of 14 units and that of the untreated dyeings of the order of 10 units.

Applying the idea of auto-oxidation, numerous examples of which are collected by Engler<sup>5</sup>, one would expect an extensive destruction of cellulose in the presence of an easily oxidizable substance. In so far as the results of the oxidation of cellulose in the presence of Ciba Blue 2B, both in the reduced and unreduced state, are concerned, this idea does not seem to be fully operative. In fact, in spite of an increased oxygen consumption with an increase in the

concentration of the dyestuff, the fluidity of the substrate is not altered (FIG. 2). The oxygen consumption appears to be simply related to the amount of dye present on the fibre. The difference of approximately 1 millimole between the reduced and unreduced dyeing (FIG. 1) appears to be mostly due to that amount required for the conversion of the leuco-Ciba Blue 2B to the fully oxidized stage.

Sharply differentiated with these results are those obtained with Cibanone Orange R (FIGS. 3 and 4). It will be seen that with an increase in the concentration of the dyestuff on cellulose the oxygen consumption also increases (FIG. 3), as was found to be the case by Nabar, Scholefield and Turner<sup>7</sup>. At the same time, the cellulose substrate has been found to have undergone considerable chemical modification (FIG. 4). For an oxygen consumption of about 3.5 millimoles (0.112 per cent), the cuprammonium fluidity of the treated dyeing has been found to be of the order of 27 reciprocal poises, indicating a considerable chemical damage. Cibanone Orange R appears to be chemically inert to hypochlorite treatment and the oxygen taken up by the reduced dyeing is used up practically in oxidizing the cellulose substrate.

In Figs. 5-8 are given results of hypochlorite oxidation experiments carried out with mixed dyeings of Ciba Blue 2B and Cibanone Orange R. It has been pointed out<sup>9</sup> that when such a mixed dyeing is

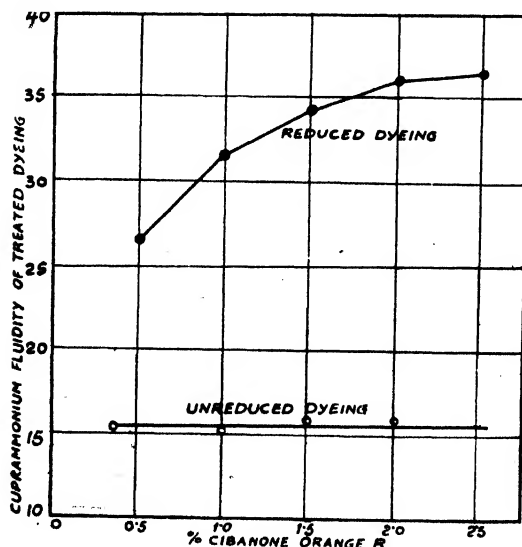


FIG. 4 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY OF THE DYEING AND THE CONCENTRATION OF CIBANONE ORANGE R.

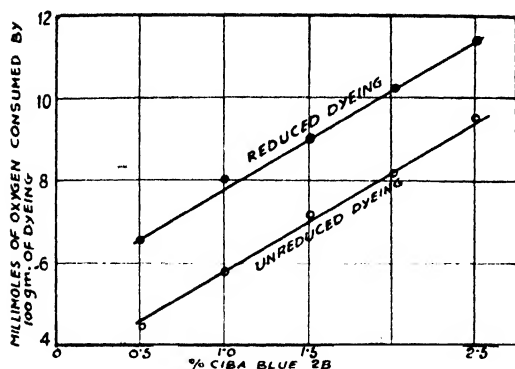


FIG. 5 — RELATION BETWEEN PERCENTAGE OF CIBA BLUE 2B AND THE AMOUNT OF OXYGEN CONSUMED BY REDUCED AND UNREDUCED DYEING IN THE MIXED DYEING.

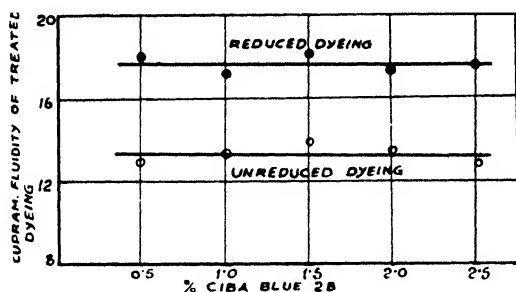


FIG. 6 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY AND CONCENTRATION OF CIBA BLUE 2B IN THE MIXED DYEING.

exposed to light and air, the blue component gets rapidly destroyed giving protection to the cellulose substrate from the degrading action of Cibanone Orange R. It is interesting to find similar behaviour to exist when mixed dyeings produced from Ciba Blue 2B and Cibanone Orange R are treated in the reduced and unreduced state with hypochlorite solutions. From Fig. 5 it will be seen that when the amount of Cibanone Orange R in the mixed dyeing is kept constant and the amounts of the Ciba Blue 2B are increased in regular amounts, the oxygen up-take also increases in the same manner. It is interesting to find that in the case of the unreduced mixed dyeings, when treated with hypochlorite solutions, the amounts of oxygen consumed are more or less the same as those consumed by the pure Ciba Blue 2B dyeings of similar concentration (Fig. 1). The amount of oxygen consumed by the reduced mixed dyeings, however, are consistently higher by about 1 millimole than those for the reduced pure Ciba Blue 2B dyeing. This consistent

difference must be due to the fact that the reduced mixed dyeing contains, in addition to Ciba Blue 2B, 2.5 per cent Cibanone Orange R, which has to be converted to the oxidized stage. Examining the cuprammonium fluidity of the treated mixed dyeings (Fig. 6), it is found that in spite of 2.5 per cent Cibanone Orange R being present on the dyeing, the rise in cuprammonium fluidity is not at all significant. When dyeing with 2.5 per cent Cibanone Orange R alone is treated in the reduced state with hypochlorite solution, a cuprammonium fluidity of the substrate rises to 36 units (Fig. 4), while the same for the mixed dyeing is of the order of 18 units (Fig. 6). The presence of Cibanone Orange R, however, causes a very small but consistent increase in the cuprammonium fluidity of the mixed samples. When these results are compared with those obtained (Figs. 7 and 8) with mixed dyeings when the concentration of Cibanone Orange R was varied, keeping that of Ciba Blue 2B at 2.5 per cent, it is seen that the increase in the concentration of Cibanone Orange R does not make any difference in the amounts of

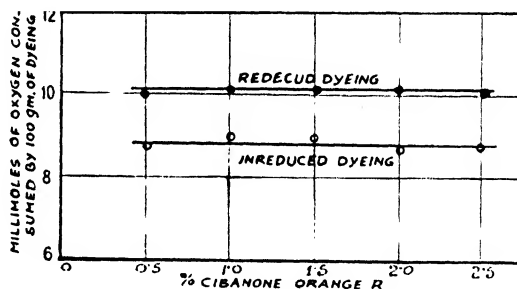


FIG. 7 — RELATION BETWEEN THE PERCENTAGE OF CIBANONE ORANGE R AND THE AMOUNT OF OXYGEN CONSUMED IN THE MIXED DYEING.

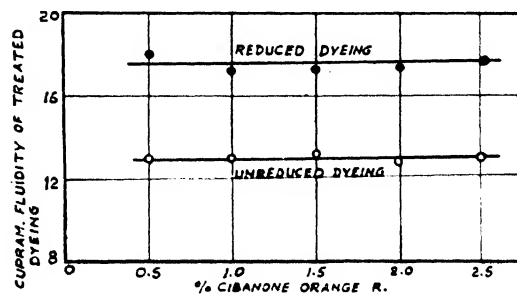


FIG. 8 — RELATION BETWEEN CUPRAMMONIUM FLUIDITY AND CONCENTRATION OF CIBANONE ORANGE R IN THE MIXED DYEING.

oxygen consumed by the mixed dyeings (FIG. 7) as was found to be the case when Cibanone Orange R alone was dyed on cotton (FIG. 3). The cuprammonium fluidity is also not altered with increase in the concentration of Cibanone Orange R. Both these observations indicate that in presence of Ciba Blue 2B, Cibanone Orange R, a pronounced tendering dye, both in the unreduced state in the presence of light and air and in the reduced state in the presence of oxidizing agents, loses completely its identity and its tendering properties appear to be lost in the presence of Ciba Blue 2B. It should be of interest to find out the mechanism by which Ciba Blue 2B renders Cibanone Orange R inactive in so far as tendering of cellulose substrate is concerned.

### Summary

1. The oxidation of cellulose dyed with Ciba Blue 2B, Cibanone Orange R and their mixture both in the reduced and unreduced state by dilute solutions of sodium hypochlorite is studied.

2. It is found that in the reduced state Ciba Blue 2B does not accelerate the oxidation of the cellulose substrate. Cibanone Orange R, on the other hand, under similar conditions causes a considerable acceleration of the oxidation of cellulose.

3. In the presence of reduced Ciba Blue 2B, reduced Cibanone Orange R loses its

identity with the result that there is no acceleration of the oxidation of cellulose by hypochlorite solutions.

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# Lac-linseed Oil Varnishes

## Part III—Lac-linseed Oil-lime

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THE possibility of preparing lac-linseed oil varnishes using lead oxide and glycerine respectively as incorporating agents has been studied and reported in two previous papers<sup>1,2</sup>. It has been pointed out that about 15 per cent of litharge on the weight of lac is necessary to ensure complete dissolution of lac in the oil. It has also been pointed out that whereas baked films of the lac-linseed oil-lead oxide compositions are quite smooth and homogeneous, air-dried films are not so smooth and present an uneven surface. This was interpreted as being possibly due to the large amount of the "drying" metal necessarily present in the composition. It was hoped, therefore, that if the lead oxide could be partially or totally replaced by the oxide of another metal of the non-drying or at least less drying type, the defect might be eliminated. With this end in view, lime (calcium hydroxide) was investigated as a possible substitute for lead oxide.

The possibility of using lime as an incorporating agent for the preparation of lac-linseed oil combinations has been reported by Aldis<sup>3</sup> and by Fain<sup>4</sup>. The former, beyond reporting one particular proportion, does not appear to have studied the problem in all its aspects such as the film properties of the resulting compositions, while the latter has used the product only in conjunction with other synthetic resins. The problem has, therefore, been investigated anew in detail. As stated already, about 15 per cent of litharge on the weight of lac is necessary to bring about proper incorporation of lac in linseed oil. This, in terms of calcium hydroxide, will be equivalent to about 5 per cent on the weight of lac. Thus, to dissolve 50 gm. of lac in 100 gm. linseed oil, 7.5 gm. of litharge or its equivalent 2.5 gm. of calcium hydroxide are required, which was actually found to be the case. For the proportions given above, the temperature of incorporation is 240°-250°C. in the case of litharge, but when lime is

used, a higher temperature (280°-290°C.) is necessary. If less than 2.5 gm. of lime are used, a portion of the lac is left behind in granular form. Increasing the amount of lime lowers the temperature of incorporation but not to the same extent as in the case of litharge. Again, when 100 parts of lac are dissolved in 100 parts of linseed oil using 15 parts litharge, the temperature of incorporation is 220°C. But with these same proportions of lac and oil and the equivalent quantity of lime (i.e. 5 parts) in place of litharge, the product obtained as a result of reaction at the same temperature, namely 220°C., does not become clear immediately as in the case of litharge but only after 20-25 min. If, however, the temperature is raised to 290°C. the product becomes clear immediately. From the above data it may be inferred that as an incorporating agent lime is not so efficient as litharge.

It was observed in the course of our experiments that precipitated calcium linoleate may be used in place of lime for incorporating lac in linseed oil (cf. lead linoleate), and it was of interest to investigate whether any other soap of calcium would serve the same purpose or it should only be the linoleate. Accordingly, calcium stearate was tried, and was found to effect the dissolution quite well. Other metallic soaps, e.g. the stearates of zinc, aluminium and potassium as well as linoleates of manganese, sodium and potassium were tried, and it was found that all of them were more or less similar in bringing about the compatibility.

Among other compounds tried were sodium acetate and sodium oleate which, however, did not induce solubility, though the "life" of lac in the oil was prolonged considerably. The chemical reactions involved in these incorporations are being examined and will be reported later.

*Properties of Lac-linseed Oil-lime Compositions*—Though lime is not quite so efficient as litharge as incorporating agent, the



products obtained by using either are very similar. Thus, lac-linseed oil-lime compositions are all soluble in the usual varnish solvents like white spirit or turpentine, and require, in addition, small proportions of alcohol to eliminate false body when the proportion of lac to oil exceeds 70-75 per cent. These varnishes are naturally slow-drying owing to the absence of lead, but when suitable driers are incorporated, the drying property can be considerably improved. The air-dried films are smooth and homogeneous. Even in the absence of a drier, the films dry when baked at 90°-95°C. in the course of 3-4 hr. and in shorter time at higher temperatures. The varnishes are non-skinning and the baked films are considerably more resistant to the effect of heat than the lead oxide compositions with regard to their ageing characteristics.

**Lac-linseed Oil-litharge-lime** — It was noted that though the lac-linseed oil-lime compositions could be made to air-dry in less than 24 hr. by using excess of lead and cobalt driers, the product could not be made to "surface-dry" in less than 6 hr. as required in some of the specifications. It was hoped that by substituting a portion of the lime with litharge, a composition might be obtained which would surface-dry in less than 6 hr. and retain all the other properties unaffected. Such a product could actually be made by using 3 parts of litharge and 1.5 parts of lime for incorporating 50 parts of lac in 100 parts of linseed oil.

A varnish based on the combined use of lime and litharge in the above proportions may be made under the usual conditions at a temperature of 270°-280°C. (as described for litharge). With the addition of 0.04 per cent cobalt in this varnish the surface-drying time is less than 6 hr. and the hard-drying time less than 18 hr. The resulting film in this case also was smooth. The acid value of the product is low and the colour satisfactory. Incidentally, this composition without the cobalt drier was found to be superior for use as a clear baking, insulating varnish to the one using only

lime as the incorporating agent, as the drying was faster. This was also perfectly non-skinning in spite of the presence of lead. A sample of "empire cloth" prepared on a 5-mil cotton support by the application of 4 coats of this varnish (by dipping), each coat being baked in a tower at 125°C. for 1 hr. gave a product with the following characteristics:

Thickness	10 mil
B.D.V. at laboratory temperature after drying in a vacuum desiccator for 24 hr.	980 volts/mil
B.D.V. at 90°C.	700-750 volts/mil

### Summary

1. Lac can be incorporated in linseed oil into which 5 per cent of lime on the weight of lac has been dissolved.
2. In place of lime the equivalent quantity of precipitated calcium linoleate or stearate may be used.
3. Other metallic soaps like zinc stearate, potassium linoleate, etc., also assist in the incorporation of lac in linseed oil.
4. Lime may be partially replaced by litharge to improve the drying characteristics of the composition.
5. The products obtained in either case are practically similar to those obtained using litharge alone except for the drying characteristics. They have, however, better ageing properties.
6. Cobalt or manganese together with lead may be used to improve the drying properties.

### Acknowledgement

The author wishes to acknowledge his grateful thanks to Dr. P. K. Bose, Director, Indian Lac Research Institute, for his interest and suggestions in the progress of the work.

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# Chemical Examination of the Dried Latex from *Euphorbia tirucalli*

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KARIMULLAH and N. L. Dutta<sup>1</sup> have reported the isolation of a crystalline substance from the dried latex of *Euphorbia tirucalli*. The present paper deals with the results of further work on the characterization of the crystalline substance, and also of the resin which constitutes the main bulk of the dried latex.

The crystalline matter was isolated in a crude form from the ether extract of the dried latex by dissolving it in a minimum quantity of hot alcohol and allowing the solution to cool. The yield of the ketone, which for ease of reference may at this stage of investigation be provisionally named *Euphoron*, varies from 20 per cent in fresh samples to 2 per cent in samples stored for about a year. In keeping with its ketonic nature, *Euphoron* gives an oxime (m.p. 194°-195°C.), a hydrazone (m.p. 149°-150°C.), and a semicarbazone (m.p. 178°-179°C.). It contains 2 double bonds as reported earlier (loc. cit.); but it takes only 2 hydrogen atoms on catalytic reduction with platinum oxide yielding a product which melts at 65.5°C. and is ketonic in character as it forms an oxime (m.p. 205°C.). Reduction with sodium and amyl alcohol, however, gave the corresponding alcohol in 20 per cent yield along with a large quantity of resinous material from which no crystalline matter could be isolated. The acetyl derivative of the alcohol, which will be referred to as *Euphorol*, melts at 107°-108°C., and is identical with the acetyl derivative of the alcohol isolated earlier (loc. cit.) from the mother liquor of *Euphoron*.

In the earlier communication, the formula  $C_{27}H_{48}O$  was proposed for the ketone on the basis of the C, H values. Although the molecular weight determined by the Rast method (M.W. 390) agrees fairly well with this molecular formula (M.W. required, 382),

the iodine value of the ketone and the nitrogen values of the oximes of the original and the hydrogenated ketones seem to be somewhat more favourable for formulations with a higher number of carbon atoms (cf. experimental). The exact formula for *Euphoron* cannot, therefore, be definitely fixed at the present stage of investigation.

The main component of the dried latex is a brittle, lustrous resin resembling rosin in appearance and melting at 65°-75°C. On distilling with superheated steam at 250°C. a portion of the resin distilled over as a viscous liquid, leaving behind a residue which melted at 125°-140°C. and was dark, lustrous and brittle in character. The linseed oil varnish prepared with this hardened resin gave films which were tack-free and glossy, indicating the possibility of its use in the formulation of paints and varnishes.

## Experimental

*Isolation of the Ketone (Euphoron)* — 5 kg. of the dried latex were powdered and exhaustively extracted with ether. The ethereal extract was concentrated to a thick syrup, which was dissolved in a minimum quantity of hot alcohol and left overnight for crystallization. The crystallizate was sucked, washed with alcohol and dried (yield, 1 kg.). On repeated crystallization of this product with chloroform, petroleum ether, ethyl acetate and alcohol, *Euphoron* was finally obtained as a colourless crystalline product melting at 119.5°C. The mother liquor yielded further quantities of the pure ketone, giving a total yield of 15 per cent on the weight of the dried latex. *Euphoron* is soluble in chloroform in the cold and ether, and in alcohol, acetone and petroleum ether in the hot and crystallizes from these solvents in star-shaped aggregates of needles.

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## ANALYSIS\*

REQUIRED FOR	C. %	H. %	M.W.	IODINE VALUE (HANUS)
$C_{27}H_{48}O$	84.80	10.99	382	132.9
$C_{29}H_{48}O$	84.87	11.22	410	123.8
$C_{31}H_{50}O$	84.92	11.41	438	116.0
Found (1)	84.57	11.37	390 (Rast)	110.0
(2)	84.72	11.28		

**Euphoron Oxime** — 1 gm. of hydroxylamine hydrochloride dissolved in 2 c.c. of water was added to 100 c.c. of alcohol containing 0.5 gm. of potassium hydroxide. The solution was then filtered and the filtrate refluxed for 1 hr. with 1 gm. of *Euphoron* on the water bath. The crystallize obtained on cooling the solution at the end of the reaction was filtered, washed with water, and dried over a porous plate. On crystallization from a mixture of alcohol and chloroform, this product gave a nearly theoretical yield of the oxime (1 gm.) which melted at 194°-195°C. and formed fine, colourless plates of crystals. *Euphoron* oxime is soluble in ether, chloroform, and ethyl acetate in the cold, and alcohol, acetone and petroleum ether in the hot.

## ANALYSIS

REQUIRED FOR	C. %	H. %	N. %
$C_{27}H_{43}ON$	81.60	10.83	3.53
$C_{29}H_{47}ON$	81.89	11.06	3.29
$C_{31}H_{51}ON$	82.12	11.26	3.09
Found	82.68	11.12	3.14

**Euphoron Semicarbazone** — To a hot solution of the ketone (1 gm.) in alcohol were added semicarbazide hydrochloride (1 gm.) and sodium acetate (1 gm.); the mixture was refluxed over a water bath for 1 hr. and then poured into water. The precipitate was washed free from sodium acetate and crystallized from alcohol. The semicarbazone thus obtained melted at 178°-179°C. and was soluble in ether, alcohol, chloroform and petroleum ether.

**Euphoron Hydrazone** — To an aqueous solution of hydrazine hydrochloride (2 gm.) was added 4 gm. of sodium acetate and 300 c.c. of alcohol and the resulting solution

\* The micro analytical values recorded in the present paper have all been determined after drying the substances to constant weight at 50°-60°C. *in vacuo*.

was refluxed with 1 gm. of ketone dissolved in a small quantity of hot alcohol for 4 hr. on the water bath. The crystals which separated on cooling the solution were filtered, washed with water and crystallized from a mixture of ether and alcohol. The hydrazone thus obtained formed pale-yellow plates readily soluble in ether, and chloroform, but sparingly so in other organic solvents; m.p. 149°-150°C.

**Dihydro Euphoron** — A solution of 5 gm. of pure *Euphoron* in 100 c.c. of glacial acetic acid was shaken with 0.5 gm. of platinum oxide catalyst (Roger-Adams) in a long-necked flask connected to a graduated hydrogen reservoir. The absorption which was rapid in the beginning was complete in 8 hr. The glacial acetic acid was distilled off from the solution below 70°C. *in vacuo*, and the residue crystallized from alcohol. Dihydro *Euphoron*, thus obtained in nearly theoretical yield (5 gm.), formed triangular plates melting at 65.5°C. and was readily soluble in chloroform, ether, alcohol and acetone. It decolourized alkaline permanganate solution and bromine in chloroform, indicating its unsaturated character.

## ANALYSIS

REQUIRED FOR	C.	H.	VOLUME OF H. ABSORBED FOR ONE DOUBLE BOND lit./100 gm.
$C_{27}H_{44}O$	84.37	11.47	5.83
$C_{29}H_{48}O$	84.47	11.65	5.44
$C_{31}H_{52}O$	84.53	11.81	5.09
Found	84.59	11.59	5.00

**Dihydro-Euphoron Oxime** — The oxime of the dihydro ketone was prepared by following the procedure described for the preparation of the oxime of *Euphoron*. It formed colourless flakes soluble in ether and chloroform, less so in alcohol and acetone, and nearly insoluble in petroleum ether; m.p. 205°C.

## ANALYSIS

REQUIRED FOR	C. %	H. %	N. %
$C_{27}H_{45}ON$	81.19	11.28	3.51
$C_{29}H_{49}ON$	81.51	11.48	3.28
$C_{31}H_{53}ON$	81.75	11.65	3.08
Found	81.40	11.45	3.28

**Euphorol\*** — A hot solution of the ketone (5 gm.) in 250 c.c. of amyl alcohol was fitted with a reflux arrangement and sodium added to it in small bits in the course of about 1 hr. After the completion of the reaction the amyl alcohol solution was washed free of alkali with water and the solvent distilled off *in vacuo*. The residue was then crystallized successively from petroleum ether, a mixture of methyl alcohol and ethyl acetate and from alcohol, when *Euphorol* was finally obtained in the form of colourless needles in a rather poor yield (1 gm.). It melts at 109°C. and is more readily soluble than the ketones in ether, alcohol and other organic solvents.

**Euphorol Acetate** — To a solution of *Euphorol* (0.5 gm.) in 10 c.c. of dry pyridine was added 5 c.c. of acetic anhydride. After warming over the water bath for half an hour, the solution was cooled, diluted with ether, and successively shaken out with dilute solutions of hydrochloric acid and sodium hydroxide, and finally with water. It was then dried over anhydrous sodium sulphate and the solvent removed on the water bath. The residue was repeatedly crystallized from alcohol when *Euphorol acetate* was obtained in the form of fine needles, readily soluble in ether and chloroform in the cold and in alcohol, acetone, and petroleum ether in the hot; m.p. 107°-108°C. (yield, 0.2 gm.).

**Euphorol Benzoate** — A solution of *Euphorol* (0.25 gm.) and benzoyl chloride (1 c.c.) in dry pyridine (10 c.c.) was warmed on the water bath for about half an hour, cooled and diluted with ether. The ethereal solution was shaken out first with a dilute solution of hydrochloric acid, then with dilute alkali, and finally with water. It was then dried over anhydrous sodium sulphate, filtered and freed from the solvent. On dissolving the residue in hot alcohol and leaving the solution overnight, *Euphorol benzoate* was obtained in colourless needles,

fairly soluble in ether and chloroform, less so in alcohol, and melting at 114°C.; yield 0.15 gm.

**Resinous Component of Dried Latex** — After separation of the total crystallizate (crude *Euphoron*) from the alcoholic solution of the ethereal extract of the dried latex, the solvent was removed from the mother liquor *in vacuo*. The residue, which constitutes the resin component of the latex, was a brittle, lustrous mass resembling rosin in appearance, and melting within the range of 65°-75°C. When distilled with superheated steam, at 250°C., it yields a small quantity of a viscous liquid. The main bulk of the resin, however, remained in the flask as a dark, brittle, lustrous mass melting at 125°-140°C. Both the steam distilled and the original resins are soluble in benzene, turpentine and linseed oil. The physical and chemical constants of the various resinous products obtained from the latex are given below.

#### ANALYSIS

	ETHER EXTRACT	TOTAL RESIN	RESIDUE AFTER TREATMENT WITH SUPERHEATED STEAM	HIGH TEMPERA- TURE STEAM DISTILLATE
Yield on the wt. of the dried latex, %	75	50	45	5
Ash, %	0.1	0.12	...	...
M.p., °C.	55-62	65-75	125-40	...
Acid value	21.6	17.7	12.3	9.1
Ester value	67.9	66.6	106.6	nil
Iodine value	113.0	131.0	55.9	62.9
Carbonyl value*	141.0	112.0	...	...

#### Acknowledgement

The authors take this opportunity to express their thanks to Dr. Weiler, Micro-analytical Laboratory, Oxford, for the analytical data incorporated in this publication.

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\* Further work on the purification of *Euphorol* will form the subject of a subsequent communication.

\* Determined by the method described by Cottrell in *Paint Technology*, 1944, 9, 73.

# An Ideal Encrustation of Lac

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**D**URING November 1923, a fresh piece of stick lac was collected at Bangalore. It belonged to the insect *Lakshadia communis* growing on *Acacia concinna*, which is a common plant there, but nevertheless only rarely attacked by the lac insect. Since no lac encrustation similar to it was collected before or subsequently, it has been the very best I ever collected. Even in the literature no record similar to it is mentioned.

I have shown elsewhere<sup>1</sup> that at the end of the monsoon season, in November, only small chunks of lac are found. From these pieces the larvae emerge as winged males. A few that get scattered, usually only 1 larva on a branch, change their sex, become crown shaped in form and give rise to a normal generation of young ones, about 13 females to 1 male. The progeny that emerges from single cells can never be very large and explains the occurrence of stick lac only as isolated chunks. The preponderance of males regularly following the monsoon season makes *L. communis* unfit for cultivation, a fact not recognized in the literature even to this day. A parallel occurrence of males in the Indian *Cochenille* insect likewise prevented its being utilized for cultivating the *Cochenille* insect in this country. I believe it is the insect originally introduced from South America where it was found wild and thus this species compares well with *L. communis* found all over South India.

When the young ones emerge from the crown-shaped cell, they settle down in the proximity of the dead mother insect. It is, therefore, commonly found that the empty cell gets incorporated with the fresh mass of encrustation produced by the living generation, as has been previously<sup>1</sup> illustrated. But the crown-shaped cell may remain apart from the main encrustation which has been likewise shown before<sup>1</sup>. The dry cell may even drop away, leaving no trace of the mother insect which has given birth to the generation forming chunk of lac. When we consider probabilities

rather than possibilities, the piece of stick lac illustrated here was formed by a generation arising from a single crown-shaped mother cell which dropped off before it was collected.

When freshly collected, it was kept in a glass jar for breeding parasites. *L. communis* in Bangalore is most liable to the attack of *Eublemma amabilis* and next to it of *Erencyrtus dewitzi*. For about 8 days no parasite was observed. When the larval swarming became intense, the piece of stick lac was removed for infecting a branch of *Guazuma tomentosa* and left there for 24 hr. and after this period replaced in the breeding cage. The larvae that swarmed from the brood lac became all winged males which was expected. Not a single parasite was reared and as far as I know no such immunity has been established before. After 6 weeks the encrustation was removed when all insect life appeared to have been dead.

In May 1947 a photograph was taken of it and illustrations were also made which are being offered to supplement it. The dry encrustation with the twig weighed 40.425 gm. It was left overnight in alcohol which dissolved the lac resin and left the twig and individual insects free. The twig was dried at 80°C. and weighed (8.092 gm.). Insects were 1,335 in number. Thus the average weight of a dry lac cell, growing in a colony and feeding during the monsoon season, was 0.0272 gm. During the dry season more lac is secreted, likewise when insects are growing isolated. A single cell of *L. communis* collected in May 1947 from *Anona squamosa* in Hyderabad weighed 0.031 gm. For *Lakshadia nagoliensis*, feeding on *Schleichera trijuga*, during monsoon season, an average dry insect and its secretion weighed 0.0188-0.0194 gm. As compared with *L. communis* the individual insects of *L. nagoliensis* were rather small but when their body surface was considered, *Nagoli* lac insects appeared to secrete more lac.

The 1,335 insects that formed the colony-encrustation were examined carefully but

indicated any injury, even in the past, from parasites. No shrivelled bodies of dead insects were observed such as would have indicated premature death. But even if 1,335 individuals had survived, it does not indicate that the progeny did not originally consist of more larvae. The generation that forms chunks of lac born of crown-shaped cells, generally consists of 13 females to 1 male. Thus for 1,335 female insects there must have been at least 126 males, so that the total larvae that issued are 1,461. Now, a normal female cell delivers 500 to 750 larvae; the crown-shaped cell, on the contrary, has been known to yield 1,500 larvae; in fact 1,505 have been actually counted. Taking 1,500 larvae as the maximum figure, 1,460 represent female and male larvae that survived to complete their life cycles, whereas 40 must have died prematurely. There is no record in the literature even to show that 1,335 individuals were derived from 1 mother cell. Such a large progeny can only come from a crown-shaped mother insect while no normal female cell can ever compete with it and literature is silent over crown-shaped cells.

The encrustation was photographed somewhat reduced and then enlarged to reproduce the object in natural size as shown in Fig. 1a. Fig. 1b gives a pen and ink outline to bring out its important features; it also represents the encrustation (natural size). Fig. 1c shows the opposite or dorsal view of the same specimen. When a twig grows vertically, larvae settle all around it; their final product, then, is a cylindrical encrustation of lac. If the twig is inclined, the population on the side facing the earth is more intense than on the other side of the twig; the final encrustation is thicker on the side facing the earth than on the other half of the twig. If the twig is still more inclined or almost horizontal, then the larvae settle only on the lower half leaving the upper surface of the twig free from colonization. Now, all grades of inclination are found and, correspondingly, lac encrustations may be perfectly cylindrical surrounding a vertical twig or may be a semi-cylindrical crust on the lower half of a horizontal one or may be any intermediate between these extremes. A glance at Fig. 1c shows that the upper end is approaching a cylinder with only a narrow line of partition between the two sides of the encrustation meeting together, while the lower end has a distinct separation with only three-fourths of the circumference

of the twig surrounded with lac. In such a small twig degrees of inclination do not explain the difference noted above. Lac insects are phototropic so that colonization is often more intense towards the end receiving more light. Such a clear difference is not apparent on studying Fig. 1b representing the side of encrustation which was facing the earth. What is specially to be noted in Fig. 1c is that the encrustation formed by rows of insects on either side of the twig shows a partition between lac from either side which has not fused, a feature special to *L. communis*. It secretes lac poorly and thus the secretion does not easily fuse.

As early as 1567 Clusius also reproduced a wood engraving where stick lac envelopes a twig without forming a cylinder. It has been reproduced as Fig. 1 of a recent communication<sup>2</sup>. The same paper shows in Fig. 3 a stick lac of *L. communis* where the two sides of an encrustation meet but do not fuse. The object illustrated here (Fig. 1c) thus supports the illustration by Clusius.

When we compare an insect belonging to *L. communis* with that of *L. nagoliensis*, the former is larger in size but, compared with its body surface, the secretion of lac is less. In simpler words, *L. communis* has a thinner coat of lac to cover its larger body while *L. nagoliensis* has a much thicker one for a smaller body. When two rows of insects belonging to *L. communis* meet, their secretions, being thin, do not fuse, which easily happens in the case of *L. nagoliensis*. The difference in stick lac of these two lac insects has been previously explained<sup>3</sup>.

It remains to add that the diameter of the twig of *Acacia concinna* in Figs. 1a, 1b, 1c was 0.6 cm. at the top, 0.7 cm. in the middle and 0.9 cm. at the basal end. Previous observations have shown that shoots with 0.75 cm. are ideal for lac infection. The present encrustation also confirms this standard.

Part of the work was done at the Research Institute, Osmania University, Hyderabad (Deccan) with the help of Mr. Kader Mohiuddin, to whom my thanks are due. Thanks are also due to Mr. Giriraj for the pen and ink drawings.

### Summary

A specimen of stick lac has been recorded from *Acacia concinna* as host, belonging to the insect *Lakshadia communis* found at



FIG. 1a



FIG. 1b

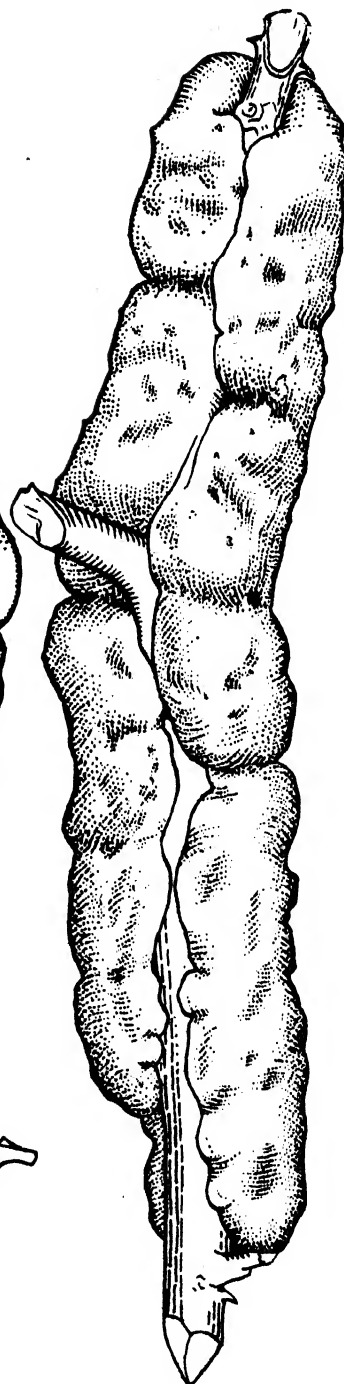


FIG. 1c

FIG. 1a—STICK LAC OF *Lakshadia communis* FORMED BY A COLONY OF 1,335 INSECTS BORN OF THE SAME MOTHER INSECT. NATURAL SIZE.

FIG. 1b—SAME AS FIG. 1a; ALSO NATURAL SIZE.

FIG. 1c—THE SAME OBJECT AS IN FIG. 1a SEEN FROM THE OTHER SIDE. THE ENCRUSTATION AT ITS BASE SHOWS A DISTINCT NARROW GAP WHILE AT THE TOP LAC SECRETION FROM THE OPPOSITE ROWS OF INSECTS IS ACTUALLY TOUCHING EACH OTHER BUT HAS NOT FUSED. NATURAL SIZE.

Bangalore, growing there during the monsoon season. 1,335 individuals formed the encrustation which together weighed 32.3 gm. The entire generation was born of one crown-shaped mother cell. The same generation also comprised probably of 126 males so that the entire progeny, including those that may have died, consisted of about 1,500 larvae. The encrustation was ideal in so far as no bigger colony, born of one

mother insect, has been recorded. It was again ideal in its being free from all parasites.

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## Letters to the Editor

### A DEPOSIT OF MASSIVE GARNET IN BIHAR SUITABLE FOR THE MANUFACTURE OF ABRASIVES & GRINDING MATERIALS

INDIA DEPENDS UPON FOREIGN COUNTRIES for the supply of materials required for cutting, polishing, crushing and grinding. Such articles are commonly required in almost all industries. Corundum sheets and papers are being manufactured on a small scale in the Mysore State. The raw material required is rare and hard to win, and a large-sized industry can be started only in a place where the raw material is available in assured commercial quantities.

A large deposit of massive garnet (calderite-manganese-iron garnet) of pinkish-black colour has been recently discovered on the bank of River Chandan near the villages Nawadih and Chandan in the Chandan sub-zemindary of the Banaili Raj, now worked by the *Mining & Industries Ltd.*, Bhagalpur. It is by the side of the Katoria-Deoghar Road and 10 miles east of Deoghar. An out-crop of  $6' \times \frac{1}{4}$  mile is visible in bold relief surrounded by eroded fields. Its development awaits the installation of an improved garnet mill as employed on the continent.

The garnet is completely free from deleterious substances, and is the purest so far discovered on so large a scale in India. The field-test for garnet to be used as abrasive is that the particles should break off and offer a fresh surface after it has been once

used. This deposit is extensive enough to warrant the establishment of a garnet mill.

Deoghar and Jasidih being the nearest railheads appear to be suitable places for this industry at present, but Jasidih being on the main line is more suitable. If the Sultanganj-Deoghar railway materializes, it will pass through this area.

Garnet-coated abrasives are used in the form of belts, discs and small sheets. 90 per cent of the garnet is used in the manufacture of abrasive-coated paper and cloth and the remainder is sold as low-priced material in the form of loose grains for surfacing and polishing marble, slate, soapstone and also in sand-blast operations. A small amount is used for the surfacing of plate glass. Garnet is also used for rubbing down painted surfaces of cars and for the manufacture of small discs employed in dental work. Garnet paper is used in leather, rubber and celluloid industries.

A. N. CHOWLA

*The Mining & Industries Ltd.*  
Bhagalpur

### FISH POISON FROM THE WOOD OF *ADINA CORDIFOLIA*

IN THE COURSE OF A STUDY OF NATURAL and synthetic insecticides and fish poisons, adinin, the crystalline component of the wood of *Adina cordifolia*, has been examined for its insecticidal and fish-poisoning properties. Adinin and its methyl ether are



found definitely toxic to fish, the methyl ether being more potent than adinin itself. It was, therefore, of interest to make a detailed study of their constitution.

Lal and Dutt<sup>1</sup> reported the isolation of a yellow colouring matter from the wood of *Adina cordifolia*, which they named "adinin". It crystallized from glacial acetic acid as small bright-yellow prisms, which on heating darkened at 195°-196°C., shrunk at 200°C. and decomposed without melting above that temperature. The molecular formula of adinin was given as  $C_{16}H_{14}O_7$ .

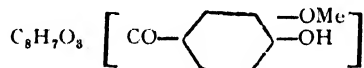
In the present work, various modifications in the procedure for the isolation and purification of adinin have been examined. Extraction of the wood material with boiling methylated spirit (repeated 3 times) brought the adinin into solution. The semi-solid residue after the removal of solvent was washed with hot water, and dissolved in acetic acid in which adinin is sparingly soluble. Other coloured impurities were eliminated by washing with petroleum ether. Precipitation from absolute alcoholic solution by the addition of a large quantity of ether gave adinin as a pale-yellow solid which melts sharply at 235°C. Analysis gave the formula  $C_{16}H_{14}O_8$  (mol. wt., by Rast's method, 294).

Adinin contains 1 methoxyl group as determined by the micro-Zeisel method. Its methylether (mol. wt. 328) has 2 methoxyl groups showing the presence of hydroxyl group. It also gives a mono-acetyl derivative. Boiling the methylether with acetic anhydride and pyridine does not bring about any change showing that adinin does not contain any other alcoholic hydroxyl group. It does not contain a sugar residue. Nor-adinin does not show any colour change in alkaline buffer solutions. The colour reactions in general seem to

indicate that adinin does not belong to the flavone group.

Adinin readily forms a dinitrophenyl hydrazone on heating with a solution of 2:4 dinitrophenyl-hydrazine in hydrochloric acid. The ease with which this derivative can be prepared shows the existence of ketonic carbonyl group.

Out of the 6 oxygen atoms in the adinin molecule, 1 is present as a methoxyl, another as a phenolic hydroxyl and the third as a carbonyl group. The nature and function of the other oxygen atoms is not yet clear. Fission of adinin methylether with 8 per cent absolute alcoholic potash and oxidation with neutral permanganate in acetone medium yielded veratric acid. This shows that the hydroxyl and methoxyl groups in the adinin molecule are present in *ortho* positions to each other in one benzene ring and this is possibly part of ketonic structure like



The location of the phenolic hydroxyl *para* to the carbonyl may account for the appreciable solubility of the compound in sodium carbonate.

Further work is under progress. The author's thanks are due to Prof. T. R. Seshadri of the Andhra University for suggesting the work and for his valuable guidance.

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*Waltair*

*June 30, 1948*

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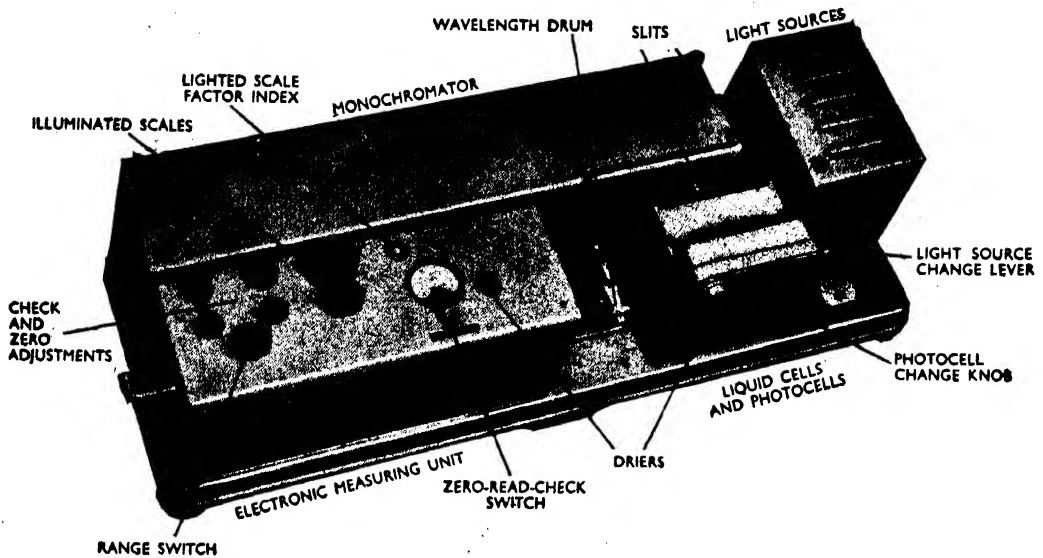
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## A Note on the Chemistry of *Solanum torvum*

G. V. KRISHNAMURTI & T. R. SESHADRI

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**S**OLANUM *torvum* is a tomentose shrub growing throughout South India. It bears white flowers and round fruits that are bitter. They are eaten as a vegetable and are considered to be of medicinal value particularly for the enlargement of liver and spleen for which no proper specifics seem to be known<sup>1</sup>. Dry *torvum* fruits are readily available in commercial quantities. Fruits of other *Solanum* species have been recently examined and new alkaloids discovered<sup>2</sup>.

The chloroform extract of the dried drug powder yields about 1.7 per cent of oily matter from which a crystalline solid melting at 248°-249°C. (decomp.) separates out in a very small quantity. It exhibits all the properties of a sterol glycoside, forms an acetate melting at 165°-166°C. and on hydrolysis yields glucose and a sterol melting at 134°-135°C. The acetate of the aglucone melts at 128°-129°C. These agree with the data given by Jantzen and Gohdes<sup>3</sup> for sitosterol-*d*-glucoside and the aglucone sitosterol. It may be recalled here that Syed and Kanga<sup>4</sup> reported the isolation of a substance melting at 248°C. from the fruit of *S. xanthocarpum*. Though they considered it to be a sterol and named it carpesterol, its solubility, high melting point and large content of oxygen would suggest that it is also in all probability a sterolin. The remaining fixed oil resembles closely in its characteristics similar oils obtained from *S. xanthocarpum* and *S. nigrum*.

The fat-free residue on extraction with alcohol yields an alkaloid which has been purified and examined. It melts at 275°-276°C. and gives characteristic reactions of the members of the *Solanum* group. On hydrolysis it yields an aglycone which is

identified as solasodine, along with glucose, rhamnose and galactose. The original glycoalkaloid should, therefore, be solasonine<sup>5</sup>. The identity has been established by comparison with a sample of glycoalkaloid isolated from *S. xanthocarpum*<sup>6</sup> and also with its products of hydrolysis. The yield of solasonine is about 0.1 per cent and the *torvum* fruit is, therefore, a comparatively poor source of the alkaloid. But this drug can more easily be obtained from *S. xanthocarpum*. The claim recorded by Kirtikar and Basu that *S. torvum* can relieve enlargement of liver is noteworthy. A similar statement has been made by Nadkarni<sup>7</sup> with regard to *S. xanthocarpum*. This property may, therefore, be mainly due to the glycoalkaloid solasonine. No detailed physiological experiment on this point seems to have been done so far.

### Experimental

Dried fruits of *Solanum torvum* were coarsely powdered. The powder (100 gm.) was successively extracted in a soxhlet with petroleum ether, chloroform, alcohol and water using 6 hours' of extraction for each solvent. Details of the various fractions are given below.

SOLVENT	YIELD OF EXTRACT	NATURE OF THE PRODUCT
Petroleum ether	1.5	Dark brown viscous liquid with aromatic odour. Mainly fixed oil with a little wax
Chloroform	0.3	Brownish-yellow waxy matter, bitter to taste
Alcohol	10.2	Brown and resinous. Very bitter. Mostly bitter material with some inorganic matter
Water	7.0	Reddish brown and astringent to taste. Mostly inorganic material along with tannin

**Sterolin & Oil** — The coarse powder (2.5 kg.) was extracted with chloroform thrice in the cold using 48 hr. for each extraction. The major bulk of the solvent was recovered by ordinary distillation and the last portion removed under reduced pressure. The residue (42.0 gm.) was viscous in consistency and dark brown in colour with a characteristic aromatic odour. On cooling and allowing it to stand for an hour, a crystalline solid separated out. It was filtered and washed with benzene. It crystallized from chloroform-alcohol mixture as shining rectangular plates and prisms melting at 248°-249°C. Yield, 0.1 gm.

The filtrate (80 c.c.) was treated with more benzene (40 c.c.) and the solution heated in a water bath with animal charcoal and filtered. The clear yellow liquid was distilled to recover most of the solvent, the last traces being removed under reduced pressure. The oil thus obtained was greenish yellow when viewed in thick layers, and bright yellow when viewed in thin layers. Yield, 40.0 gm.

The crystalline solid melting at 248°-249°C. was sparingly soluble in chloroform, moderately in alcohol and insoluble in ether. It was tasteless and responded to Molisch test for carbohydrates and to Libermann-Burchard reaction for sterols. Hence it was a sterolin. On acetylation with acetic anhydride and a few drops of pyridine and crystallizing the product from absolute alcohol, the acetyl derivative was obtained in the form of colourless, shining needles melting at 165°-166°C. When the sterolin was hydrolysed with aqueous alcoholic sulphuric acid and the aglucone (sterol) crystallized from alcohol, it was obtained as colourless, shining, rectangular plates melting at 134°-135°C. The acetate of the aglucone melted at 127°-128°C. The acid filtrate containing the sugar was neutralized with barium carbonate, filtered and the filtrate concentrated to small bulk. The syrup yielded glucosazone on treatment with excess of phenyl hydrazine reagent (cf. Jantzen and Gohdes<sup>3</sup> for sitosterol-*d*-glucoside).

The oil obtained was an yellow, viscous liquid with a fine aromatic odour and burning with a smoky flame. It did not solidify on cooling even up to -5°C. but only became thick. The following are the properties of the oil: acid number, 10.4; saponification number, 183.5; Hehner number, 90.8; unsaponifiable matter, 2.3 per

cent; iodine number, 113.5; mean mol. wt. of fatty acids, 298.6. There is close agreement between this oil and those obtained from *S. xanthocarpum*<sup>8</sup> and *S. nigrum*<sup>9</sup>.

**Glycoalkaloid** — The defatted fruit was then extracted with cold alcohol thrice using 2 days for each extraction. The combined alcoholic extract was distilled to recover most of the solvent, the last portions being removed by evaporation on a water bath. The brown residue was extracted with warm water containing a little acetic acid (250 c.c.). The aqueous extract was cooled and shaken with ether to remove waxy and oily impurities. The clear aqueous solution was brought to boiling and dilute ammonia added when a granular dark-yellow solid separated out. It was allowed to settle and the supernatant liquid decanted. The precipitate was dissolved in dilute acetic acid (5 per cent) and reprecipitated by ammonia. The process was repeated twice. Finally it was crystallized from ethyl alcohol (60 per cent) first and then from methyl alcohol when it separated as long, shining needles melting at 275°-276°C. Yield, 2.0 gm.

It had a marked bitter taste. It was soluble in chloroform, alcohol and water and insoluble in ether, benzene and petroleum ether. It contained nitrogen and gave a precipitate with Mayer's reagent. It dissolved in strong sulphuric acid forming an yellow solution changing to orange-red and finally brown. It gave mild frothing with water and a positive test for carbohydrates. The glycoalkaloid was hydrolysed with sulphuric acid (7 per cent) and filtered. The filtrate was examined for sugars and the presence of glucose and rhamnose was established by the preparation of their osazones and that of galactose by conversion into mucic acid. The precipitate of aglycone salt was washed free of acid, crystallized from alcohol and boiled with dilute ammonia to liberate the free base. The mixture was filtered hot and the precipitate was washed and crystallized from alcohol when it came out as shining, star-shaped, white plates melting at 200°-202°C. (found: C, 75.3; H, 10.7;  $C_{27}H_{48}O_2N$ ,  $H_2O$  requires C, 75.2; H, 10.4 per cent). The aglycone was insoluble in petroleum ether and cold ether and soluble in hot ether, chloroform and alcohol. It dissolved in strong sulphuric acid forming a red solution, turning brown on keeping. When an alcoholic solution of the substance was

treated with sulphuric acid, green fluorescence characteristic of solasodine was produced. Further it gave a series of colour reactions with anisaldehyde and acetic-sulphuric acid. The acetyl derivative of the aglycone was obtained in the form of narrow plates melting at 195°-196°C. On refluxing with methyl iodide and crystallizing the separated solid, the aglycone formed a hydriodide melting at 283°-284°C. (found: C, 59.9; H, 8.2;  $C_{27}H_{43}O_2N$ , HI requires C, 59.9; H, 8.1 per cent). It also formed "azo-solasodine" melting at 260°-261°C. and a picrate melting at 144°-145°C.

### Summary

The dried fruit of *S. torvum* has been examined in detail. The chloroform extract yields a small amount of a sterolin (sitosterol-*d*-glucoside) and 1.7 per cent of a fatty oil which resembles closely similar oil obtained from *S. xanthocarpum* and

*S. nigrum*. Subsequent alcoholic extract gives an yield of 0.1 per cent of a glycoalkaloid which could be identified as solasonine by a detailed study of its properties and reactions and of the products of hydrolysis. The identification has been confirmed by direct comparison of the glycoalkaloid and its hydrolytic products with those obtained from *S. xanthocarpum*.

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## Potassium Chromate as a Deleading Agent for the Determination of Clerget's Sucrose

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THE method of double polarization for the determination of sucrose was first suggested by Biot; according to Brown<sup>1</sup>, this method was further developed by Clerget, who specified the length of the observation tube and the temperature at which polarization readings are to be taken. Much literature has since accumulated indicating the complexity of this determination; new modifications have been suggested, and the influence of optically active bodies and of reversion and condensation products, either present in the sugarcane or formed during the decomposition of fructose and other unstable bodies has been examined.

Among the more important modifications of determining Clerget's sucrose, mention may be made of deleading with (a) anhydrous

sodium carbonate; (b) potassium oxalate; and (c) sodium phosphate. Noel Deerr<sup>2</sup> developed the neutral polarization method employing equivalent quantities of barium hydroxide, aluminium sulphate and sulphuric acid. Based on similar considerations, and keeping in view the influence of the acid on invert polarization, Jackson and Gillis<sup>3</sup>, in their method IV, proposed the addition of sodium chloride solution (a neutral salt) to the direct polarization liquid. The International Committee on Uniform Methods of Sugar Analysis tentatively recommended Jackson-Gillis' method IV as the standard for adoption.

Zerban<sup>4</sup> re-examined the question of double polarization of sugar products and found that Jackson-Gillis' method IV gives correct values only with pure sugars, either

alone or in the presence of reducing sugars, and that the method fails when amides, amino acids and reversion and condensation products were present, the only reliable method in such cases being yeast inversion.

In view of the importance of sucrose determination to the sugar industry, the method of determining Clerget's sucrose was re-examined from the stand-point of complications introduced by lead and deleading agents on polarization. A new and simple method of deleading with potassium chromate, excess of which can be exactly controlled by using silver nitrate solution as an external indicator, has been devised and tested.

Sucrose is calculated from the general equation,\*

$$S = \frac{100 (D - I)}{143.2 - 0.0676 (m - 13) - \frac{T}{2}}$$

employing half normal solutions, and Clerget's constants were obtained by reference to Table 15 of *Methods of Chemical Control, Hawaiian Sugar Technologist's Association* 1931 (p. 130). For sucrose estimation by yeast inversion, 10 c.c. of 5 per cent *B.D.H.* yeast solution preserved with toluene were used.

### Experimental

*Influence of Deleading Agents on the Polarization of Sugar Solutions*—The use of anhydrous sodium carbonate, sodium phosphate and potassium oxalate for deleading sugar solutions prior to direct polarization and inversion is a common practice. Incomplete deleading leads to the precipitation of lead as lead chloride in the inversion flask and introduces a precipitate error. To avoid this, complete deleading is essential, and an excess of deleading agent is usually present in sugar solutions, as there is no simple means for the precise control of deleading. The presence of deleading agents interferes with direct polarization, as may be seen from Table I.

It will be seen that excess of deleading agents introduces errors in the results. The

TABLE I

50 c.c. of sugar solution + deleading agent in 3 different quantities, viz. 0.5 gm., 1.5 gm. and 2.5 gm.; solution made up to 100 c.c. and polarized. Figures indicate direct polarization readings

DELEADING AGENT	POLARIZATION READINGS			
	Blank	0.5 gm.	1.5 gm.	2.5 gm.
1. 15% sugar solution + 0.5% glucose by vol.				
Sod. phosphate	28.5	28.7	28.2	28.9
Pot. oxalate	28.5	28.8	29.7	28.0
Sod. carbonate	28.5	26.7	27.0	26.2
2. 15% sugar solution + 1.0% glucose by vol.				
Sod. phosphate	29.5	29.8	29.2	29.0
Pot. oxalate	29.5	29.3	30.5	29.9
Sod. carbonate	29.5	30.5	30.2	30.1

TABLE II—CLARIFICATION WITH DRY POWDERED ALUMINIUM HYDROXIDE (Vol. of solution 50 c.c.)

SOLUTION	DIRECT POL.	INVERT POL.	CLERGET'S SUCROSE
1. 5 gm. sucrose	9.9 Clarified with al. hydroxide	...	...
	0.9 Unclarified	...	...
2. 5 gm. sucrose + 0.25 gm. glucose + 0.25 gm. fructose	10.3 Clarified with al. hydroxide	1.4	9.28
	10.3 Unclarified	1.4	9.28
3. 5 gm. sucrose + 0.25 gm. of aspartic acid + 0.5 gm. of asparagin	9.3 Clarified with al. hydroxide	1.4	8.43
	9.3 Unclarified	1.4	8.43

usefulness of a new deleading agent, viz. potassium chromate, was investigated as its adoption lends itself to exact control. The results obtained by the use of potassium chromate have been compared with those obtained by Jackson-Gillis' method and by using dry powdered aluminium hydroxide as clarifying agent. That the use of aluminium hydroxide does not introduce any errors while working with pure substances has been repeatedly confirmed. Table II gives one set of such results. From the table it can be seen that the values obtained for direct and invert polarimetric readings and Clerget's sucrose are exactly identical where the solutions are clarified with dry powdered aluminium hydroxide and where no clarifier is employed. Sugar solutions containing different quantities of glucose and water-soluble ash from molasses were prepared, and the sucrose determined by Jackson-Gillis' method. The results obtained are given in Table III.

It will be seen from the table that the results obtained by deleading with potassium chromate correspond to the values obtained by aluminium hydroxide clarification, indicating that for a wide variation in the concentration of glucose and ash, deleading with chromate gives satisfactory

\* S stands for sucrose percentage (Clerget); D, direct polarization reading; I, invert polarization reading; T, temperature of invert solution in centigrade; m, normal weight of sugar product, 26 gm. in 100 gm. of solution. In all the calculations half normal weights are taken so that  $0.0676(m - 13)$  cancels itself.

TABLE III

SUGAR SOLUTION	SUCROSE DETERMINED				
	Al. hydroxide	Pot. chromate	Sod. phosphate	Pot. oxalate	Sod. carbonate
20% sucrose solution + $\frac{1}{2}$ % glucose by vol.	18.45	18.45	18.52	18.52	18.57
20% sucrose solution + $\frac{1}{2}$ % ash by vol.	18.61	18.61	18.01	18.01	18.42
20% sucrose solution + 1% glucose by vol.	19.19	18.95	18.60	18.60	18.57
20% sucrose solution + 1% ash by vol.	18.51	18.51	18.08	18.08	18.21
20% sucrose solution + 2% glucose by vol.	19.04	19.04	18.73	18.73	18.57
20% sucrose solution + 2% ash by vol.	17.87	17.89	17.48	17.48	17.79
20% sucrose solution + 3% glucose by vol.	18.57	18.57	17.89	17.98	18.13
20% sucrose solution + 3% ash by vol.	18.35	18.27	18.0	18.0	17.96

TABLE IV — DIRECT POLARIZATION OF SOLUTIONS AFTER DELEADING WITH DIFFERENT DELEADING AGENTS

HORNE'S DRY LEAD ADDED TO 100 C.C.	AL. HYDROXIDE	SOD. PHOSPHATE	POT. CHROMATE	POT. OXALATE	SOD. CARBONATE
1. 10% sucrose + 0.5% glucose solution by vol.					
0.63 gm.	38.8	39.0	38.7	38.6	38.6
1.25 "	38.8	39.0	38.7	38.5	38.6
1.88 "	38.8	39.0	38.8	38.3	38.3
2. 10% sucrose + 1% glucose solution by vol.					
0.63 gm.	41.2	41.3	41.2	41.2	41.1
1.25 "	41.2	41.1	41.2	41.0	41.1
1.88 "	41.2	41.1	41.3	41.0	41.0
3. 10% sucrose + 2.0% glucose solution by vol.					
0.63 gm.	44.1	43.3	44.1	43.7	43.6
1.25 "	44.1	43.0	44.1	43.8	43.0
1.88 "	44.1	43.1	44.1	43.7	42.2

results. Further, while the actual variation in the final values recorded in Table III appears small, the variation in readings of direct polarization are considerable. This will be clear from the readings in Table IV.

Deleading with potassium chromate using test solutions containing different concentrations of glucose and excess of lead does not introduce any errors in direct polarization while the use of other deleading agents leads to errors, especially as the concentration of glucose increases.

Even though fructose as such is not known to exist except in products from unripe sugarcane, experimental solutions containing fructose were prepared and the effect of deleading agents studied. The results (TABLE V) indicate that sucrose values obtained with potassium chromate

and aluminium hydroxide clarification closely agree.

*Effect of Amides & Amino Acids* — Though present in small quantities, asparagine and aspartic acid are known to influence the values for sucrose as determined by Clerget's procedure. The amino compounds are completely removed by deleading with potassium chromate, while they are unaffected when other deleading agents are employed (TABLE VI).

It is clear that the use of potassium chromate eliminates the errors following the use of sodium phosphate, potassium oxalate and sodium carbonate in deleading sugar solutions containing glucose, fructose, amides and amino acids and ash-forming materials. The method has been further compared with Jackson-Gillis' method IV for the analysis of cane juices. The comparative data are presented in Table VII.

TABLE V

SOLUTION	CLERGET'S SUCROSE %				
	Al. hydroxide	Pot. chromate	Sod. phosphate	Pot. oxalate	Sod. carbonate
10 gm. sucrose + 0.5 gm. fructose in 100 c.c. solution	9.41	9.34	8.6	9.0	9.0
10 gm. sucrose + 1.0 gm. fructose in 100 c.c. solution	9.8	9.8	9.8	9.5	9.3

TABLE VI — EFFECT OF AMINO ACIDS

50 c.c. sucrose solution, 10 c.c. 0.25% asparagine or 10 c.c. 1.0% aspartic acid or 10 c.c. each of both treated with lead, deleading and made up to 100 c.c. and polarized.

DELEADING AGENT	POLARIZATION READINGS	
	Control	Experimental
1. 15% sucrose solution + 10 c.c. 0.25% aspartic acid by vol.		
Pot. chromate	28.7	28.7
Sod. phosphate	28.7	28.4
Pot. oxalate	28.7	28.4
Sod. carbonate	28.7	28.1
2. 15% sucrose solution + 10 c.c. 1% asparagine by vol.		
Pot. chromate	28.5	28.5
Sod. phosphate	28.5	28.0
Pot. oxalate	28.5	28.1
Sod. carbonate	28.5	28.0
3. 20% sucrose solution + 0.25% fructose + 1.75% glucose 1% asparagine by vol.		
Pot. chromate	38.8	38.8
Sod. phosphate	38.8	38.4
Pot. oxalate	38.8	38.5
Sod. carbonate	38.8	37.3
4. 20% sucrose solution + 0.25% fructose + 1.75% glucose + 10 c.c. 0.25% aspartic acid		
Pot. chromate	38.0	38.0
Sod. phosphate	38.0	38.6
Pot. oxalate	38.0	38.7
Sod. carbonate	38.0	38.7



It is seen from the above that Jackson-Gillis' method and chromate deleading followed by acid or yeast inversion give closely agreeing results both for raw and clarified juices taken from factory samples.

*Determination of added Sucrose in Molasses & Vinasses*—The application of this method has been extended to the estimation of added sucrose to molasses and vinasses. The results obtained are shown in Tables VIIIA, VIIIB, IXA and IXB.

TABLE VII

	CLERGET'S SUCROSE %		
	Jackson-Gillis' method	Pot. chromate, yeast inversion	Pot. chromate, acid inversion
1. Raw juice	12.42	12.48	12.30
Clarified juice	14.61	14.72	14.54
2. Raw juice	10.23	10.35	10.34
Clarified juice	12.75	12.79	12.78
3. Raw juice	11.69	11.76	11.74
Clarified juice	13.90	13.94	13.94

TABLE VIIIA—ESTIMATION OF SUCROSE ADDED TO EXHAUSTED MOLASSES

Method of deleading	ACID INVERSION		
	Sugar added	Sugar recovered	Variation
Jackson-Gillis	7.63	4.21	-3.42
	11.44	10.98	-0.46
	15.24	17.03	+1.79
	19.06	16.28	-2.78
Pot. chromate	7.63	7.83	+0.20
	11.44	11.9	+0.46
	15.24	15.03	-0.21
	19.06	19.03	-0.03
Pot. oxalate	7.63	9.52	+1.89
	11.44	9.4	-2.02
	15.24	12.56	-2.68
	19.06	18.11	-0.95
Sod. carbonate	7.63	4.11	-3.52
	11.44	11.9	+0.46
	15.24	12.56	-2.68
	19.06	15.06	-3.4
Sod. phosphate	7.63	7.21	-0.42
	11.44	10.05	-1.39
	15.24	17.04	+1.8
	19.06	19.52	0.46

TABLE VIIIB—ESTIMATION OF SUCROSE ADDED TO EXHAUSTED MOLASSES

Method of analysis	YEAST INVERSION		
	Sugar added	Sugar recovered	Variation
Pot. chromate, acid inversion	3.8	3.78	-0.02
	7.63	7.64	+0.01
	15.11	15.01	-0.1
Pot. chromate, yeast inversion	3.8	3.81	+0.01
	7.63	7.75	+0.21
	15.11	15.00	-0.11
Herzfeld-yeast inversion	3.8	4.4	+0.6
	7.63	7.77	+0.14
	15.11	15.01	-0.1

TABLE IXA—ESTIMATION OF SUCROSE ADDED TO DISTILLERY VINASSES BY DIFFERENT METHODS OF ANALYSIS

METHOD	SUGAR ADDED	SUGAR RECOVERED	VARIATION
1. Sod. phosphate, acid inversion	15.26	18.07	+3.81
	19.12	16.56	-2.56
	22.93	21.82	-1.11
2. Pot. oxalate, acid inversion	15.26	13.59	-1.67
	19.12	20.22	+1.10
	22.93	22.29	-0.63
3. Sod. carbonate, acid inversion	15.26	14.06	-1.20
	19.12	21.16	+2.04
	22.93	24.03	+1.10
4. Sod. carbonate, yeast inversion	15.26	15.35	+0.09
	19.12	19.11	-0.01
	22.93	22.70	-0.14
5. Pot. chromate, acid inversion	15.26	15.36	+0.10
	19.12	19.11	-0.01
	22.93	22.79	-0.14
6. Pot. chromate, yeast inversion	15.26	15.36	+0.10
	19.12	19.14	+0.02
	22.93	22.70	-0.14
7. Jackson-Gillis	15.26	16.76	+1.52
	19.12	20.54	+1.42
	22.93	25.07	+2.14

TABLE IXB—ESTIMATION OF SUCROSE ADDED TO DISTILLERY VINASSES IN THE PRESENCE OF AMINO COMPOUNDS

1.0 gm. asparagine and 0.22 gm. aspartic acid in 100 c.c. solution			
METHOD	SUGAR ADDED	SUGAR RECOVERED	VARIATION
1. Sod. phosphate, acid inversion	15.26	16.92	+1.66
	19.12	18.28	-0.84
	22.93	23.63	+0.70
2. Pot. oxalate, acid inversion	15.26	14.21	-1.05
	19.12	16.69	-2.43
	22.93	24.11	+1.18
3. Sod. carbonate, acid inversion	15.26	16.74	+1.48
	19.12	17.70	-1.33
	22.93	21.39	-1.54
4. Sod. carbonate, yeast inversion	15.26	15.48	+0.22
	19.12	19.06	-0.06
	22.93	22.70	-0.23
5. Pot. chromate, acid inversion	15.26	15.32	+0.06
	19.12	19.10	-0.02
	22.93	22.70	-0.23
6. Pot. chromate, yeast inversion	15.26	15.32	+0.06
	19.12	19.10	-0.02
	22.93	22.70	-0.23
7. Jackson-Gillis	15.26	17.71	+2.45
	19.12	19.87	+0.75
	22.93	22.08	-0.85

According to Saillard<sup>5</sup>, the suitability of a method for sucrose estimation can be assessed by studying the effects of pre-existing salts and nitrogenous bodies in molasses on the polarization of sucrose. For this purpose, distillery vinasses containing these non-sugars but little or no sugar was taken, known quantities of sugar added and the solution analysed for Clerget's

sucrose by different methods. In Table IXA are given the results obtained by applying the chromate method. In Table IXB are given the results obtained when the methods are applied to determine sucrose added to vinasses, asparagine and aspartic acid being also added.

The results indicate that Jackson-Gillis' method gives results which depart from the true values, while the chromate-yeast, chromate-acid and Herzfeld-yeast inversion methods give correct values for sucrose within permissible limits of experimental error.

The above studies show that deleading with potassium chromate provides a

satisfactory means of eliminating the errors due to the presence of ash constituents, amides and amino acids normally present in molasses and distillery vinasses.

The authors record their thanks to Dr. B. Viswanath for suggesting this investigation.

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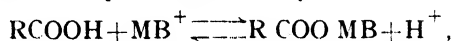
## Some Properties of Acidic Oxycellulose

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**C**ELLULOSE when oxidized gives rise to degradation products of varying properties<sup>1</sup>. It is believed that the oxidizing agents convert the hydroxyl groups in the cellulose molecule either to the aldehydic or to the carboxylic acid stage or to a mixture of both. The products of oxidative degradation, therefore, are generally more acidic in character and also possess more reducing power than the original cellulose. The high affinity of oxycelluloses for basic dyes or for metal ions has been attributed to the presence of carboxylic acid groups. A qualitative method of estimating the extent of carboxylic acid groups formed in oxycelluloses depends on the measurement of the absorption of methylene blue by the sample<sup>2</sup>. Recently, a number of methods for the direct estimation of carboxylic acid groups in cellulose have been suggested<sup>3-6</sup>. Of these methods, the one of Neale and Stringfellow<sup>3</sup> appears to be more convenient and easy to work. Since high methylene blue absorption is accompanied by high carboxylic acid content,

it is considered that the two quantities bear a relationship as indicated by



where  $\text{---COOH}$  represents the carboxyl group of the cellulose material and MB represents the methylene blue ion in the dye solution. The present investigation was undertaken with a view to see to what extent these two quantities were related to one another.

In a recent communication, Davidson<sup>7</sup> has stressed the importance of the methylene blue absorption in determining the carboxylic acid content of oxycelluloses. After a systematic study of the effect of various conditions such as the pH of the methylene blue solution, the influence of various ions present, etc., he has come to the conclusion that the methylene blue absorption values give a direct measure of the carboxylic acid content, provided the measurement is carried out under strictly controlled conditions. His results show that the value of carboxylic acid groups as estimated by the methylene blue absorption are lower than

those obtained by the alkali titration method<sup>3</sup>, the divergence between the two being more marked for oxycelluloses having higher reducing properties. The present investigation was undertaken independently of the one referred to above and the results set out in the following indicate a good amount of agreement with the conclusions arrived at by Davidson<sup>7</sup>.

Oxycellulose samples were prepared using different oxidizing agents such as sodium hypochlorite, potassium permanganate, potassium dichromate, sodium hypobromite, hydrogen peroxide, nitrogen dioxide, etc. The oxidation experiments were carried out under different conditions such as the concentration of the active oxidant, the hydrogen ion concentration, the time of treatment of the material with the oxidizing agent and oxygen consumption. Other samples such as those prepared by the action of heat and light were also examined. The absorption of methylene blue by these oxycellulose samples was carried out by the method developed by the B.C.I.R.A.<sup>2</sup> and the amount of carboxylic acid was estimated by the method of Neale and Stringfellow<sup>8</sup>. As a result of these investigations the following conclusions have been arrived at: (a) the methylene blue absorption values and the carboxylic acid values do not bear any *direct* relationship with one another. However, they appear to depend on one another; (b) the variation in the ratio between these two quantities is more marked for oxycellulose samples having higher reducing property; and (c) the methylene blue absorption values are considerably affected by the ratio of the amount of cotton to the methylene blue solution and the concentration of the latter.

### Experimental

The cotton used for the preparation of oxycellulose samples was 14's single yarn manufactured from Indian cotton. The yarn was freed from impurities by the following treatment:

The yarn was steeped in water overnight and after squeezing was boiled under pressure (20 lb./sq. in.) for 4 hr. using 2 per cent caustic soda and 0.25 per cent Lissapol C on the weight of the material. At the end of the boil the material was washed and the boiling treatment was repeated. The boiled yarn was washed free from alkali and then treated for 1 hr. with a freshly prepared bleaching powder solution containing 0.5 gm.

of available chlorine per litre. The yarn was washed free from chlorine and then scoured in 0.25 per cent hydrochloric acid solution. It was finally washed free from acid, dried and carefully stored.

### Preparation of Oxycellulose Samples

**Hypochlorite Samples** — Purified cotton in the form of hanks was immersed in sodium hypochlorite solution of approximately 3 gm. available chlorine per litre with a material liquor ratio 1:20. The pH of the solution was varied using suitable buffer mixtures.

The treatment was carried out at 30°C. with solutions having different pH. In one set of experiments the time of treatment was kept constant; in another set, the oxidation was carried out in order to obtain approximately the same oxygen consumption. Influence of the variation of the strength of active chlorine was also examined. The active strength of the hypochlorite solution used for the experiments was estimated iodimetrically.

**Permanganate Samples** — Cotton hanks were treated with 0.04N potassium permanganate solution with a material liquor ratio 1:20 at different pH values for such period as to get the same oxygen consumption and also for a constant period so as to vary the oxygen consumption. The active strength of permanganate was measured as described above.

**Hypobromite Samples** — These samples were prepared exactly as described for hypochlorite using N/10 sodium hypobromite solution at different pH values. The sodium hypobromite solution was prepared by acidifying a mixture of potassium bromide and potassium bromate and neutralizing the same with caustic soda<sup>1</sup>.

**Acid Dichromate Samples** — The samples were prepared as described above using dichromate in sulphuric acid for different strengths of dichromate and sulphuric acid.

**Nitrogen Peroxide Samples** — The oxidation was carried out in a bottle (10 litre capacity). It was fitted with a ground-in stopper, carrying a glass hook from which the sample to be oxidized was suspended in the bottle. The bottle was first dried by blowing hot air. After cooling it was evacuated to 25" vacuum. The evacuated bottle was filled with nitrogen peroxide.

**Hydrogen Peroxide Samples** — The yarn was immersed in N/5 hydrogen peroxide solution adjusted to different pH values.

The reaction flasks were kept in water bath maintained at 80°C.

At the end of each oxidation treatment, the samples were removed from the reaction flask and freed from impurities by repeated washings with water. In the case of permanganate samples, the washed samples were treated with acidified sodium bisulphite solution so as to remove the deposited oxides of manganese, then washed thoroughly free from impurities. The dichromate samples, even after thorough washing, showed a bluish-green tint due to fixed chromium which could not be removed even after acid treatment.

All the oxycellulose samples were then treated with dilute hydrochloric acid in order to free the carboxylic acid groups. They were then washed free from acid with distilled water, dried, conditioned and stored away from dust.

**Buffer System Employed<sup>8</sup>** — Mixtures of M/15  $\text{KH}_2\text{PO}_4$  and M/15  $\text{Na}_2\text{HPO}_4$  for pH values from 5.2 to 8; mixtures of M/5 acetic acid and M/5 sodium acetate for pH values from 4 to 5; M/5 acetic acid for pH 2.7; N/10 sulphuric acid for pH 1; M/20 borax for pH 9.10; mixture of M/20 borax and M/10 sodium carbonate for pH 10; M/10 sodium carbonate solution for pH value of nearly 11; M/100 sodium hydroxide for pH 12 and M/10 sodium hydroxide for pH 13.

**Determination of Carboxyl Content in Cellulose by Alkali Titration Method<sup>3</sup>** — 1 gm. of the air-dried material (allowing for hygroscopic moisture) was weighed in a 250 c.c. stoppered conical pyrex flask and treated with 50 c.c. of N hydrochloric acid for half an hour. It was then washed with distilled water followed by washing with carbon dioxide-free distilled water until the wash waters were neutral to bromo-cresol purple (pH 5.2 to 6.8). The cotton was squeezed with a glass rod and 20 c.c. of pure sodium chloride solution (5 per cent) was added. This was followed by an addition of 6 drops of bromo-cresol purple and 20 c.c. of N/50 carbonate-free caustic soda solution and 20 c.c. of carbon dioxide-free distilled water. Carbon dioxide-free air was then bubbled through the mixture for 10 min. and the flask stoppered. At the end of 1 hr., the contents of the flask were titrated against N/50 hydrochloric acid. A blank in absence of cotton was carried out. From the difference in the readings the amount of alkali consumed by the carboxyl groups

of the cellulose material was calculated. Results are expressed as milli-equivalents of carboxyl groups per 100 gm. of cotton.

**Determination of Copper Number** — The copper number determinations were carried out by the Schwalbe-Braidy method<sup>9</sup> as modified to use smaller quantities of cotton by Hayes<sup>10</sup>. In place of N/25 permanganate, N/50 ceric sulphate was employed for titration of the ferrous iron formed, using *ortho*-ferrous-phenanthroline as internal indicator. As many of the samples had very high copper numbers, smaller quantities of cotton instead of those recommended had to be used.

**Determination of Methylene Blue Absorption** — The methylene blue employed in the experiments was the pure crystalline sample which was prepared by crystallizing commercial methylene blue hydrochloride from absolute alcohol. Its strength was estimated by the following methods: (1) by titrating against titanous chloride; (2) by determining the nitrogen and sulphur content; (3) gravimetrically estimating its perchlorate<sup>11</sup>; and (4) by titrating against standard Naphthol Yellow S solution. Naphthol Yellow S was purified by crystallizing it 3 times from aqueous alcohol and its purity estimated by determining the nitrogen content. The methylene blue absorption was carried out according to the standard method as given by Birtwell, Clibbens and Ridge<sup>2</sup>. It consists in treating 2.5 gm. of cotton in 15 c.c. M/250 methylene blue solution for 18 hr. at constant temperature and quantitatively determining the absorption of methylene blue from the difference in the titration readings before and after absorption against standard Naphthol Yellow S solution. As the pH of the methylene blue brings about variation in the absorption, the methylene blue solution was buffered at pH 7. It was found that using 15 c.c. of M/250 methylene blue solution for 2.5 gm. of cotton was not adequate for a number of oxycellulose samples under examination. Hence, the conditions of experiments were modified both by increasing the quantity of methylene blue solution and by decreasing the amount of cotton used for absorption. It was, however, found that by changing the ratio of the methylene blue solution to cotton, the methylene blue absorption values as obtained were found not comparable with one another. Hence, a study of the factors influencing the methylene blue absorption values was carried out using a fairly degraded cellulose sample.

In one set of experiments, 25 c.c. of M/250 methylene blue solution was taken and the amount of the cotton was varied. In the second set, 0.5 gm. of cotton was taken in each case and the volume of M/250 solution was varied. In the third set, the volume was kept constant at 15 c.c. and the amount of cotton also kept constant at 0.5 gm. but the concentration of the methylene blue solution was varied. Finally, in the fourth set of experiments, a large excess of methylene blue solution over the quantity likely to be absorbed by cotton was taken and varying amounts of cotton were immersed in this solution. In order that the yarn should properly mix with the solution, the

stoppered bottles containing the samples were tied to a cycle wheel which was kept rotating throughout the 18 hours at about 4 revolutions per minute. Though the oxycellulose sample in all the above sets of experiments was the same, the methylene blue absorption values were different. This is clearly seen from Table I.

### Results

The results of these investigations have been summarized in Tables I to VII and shown graphically in Figs. 1 and 2. Fig. 1 represents the relation between methylene blue absorption and carboxylic acid content of oxycellulose obtained with sodium hypochlorite and potassium permanganate. Fig. 2 represents similar relationship for oxycellulose samples obtained with sodium hypobromite, hydrogen peroxide, acid dichromate, heat and light and a few samples with permanganate solutions.

In Table II are given results obtained with hypochlorite oxidized samples. From a close examination of the copper number and the corresponding ratio ( $\text{COOH}/\text{M.B.}$ ) it is seen that the value of the ratio is very nearly unity for samples having copper numbers below 1.2. For copper numbers

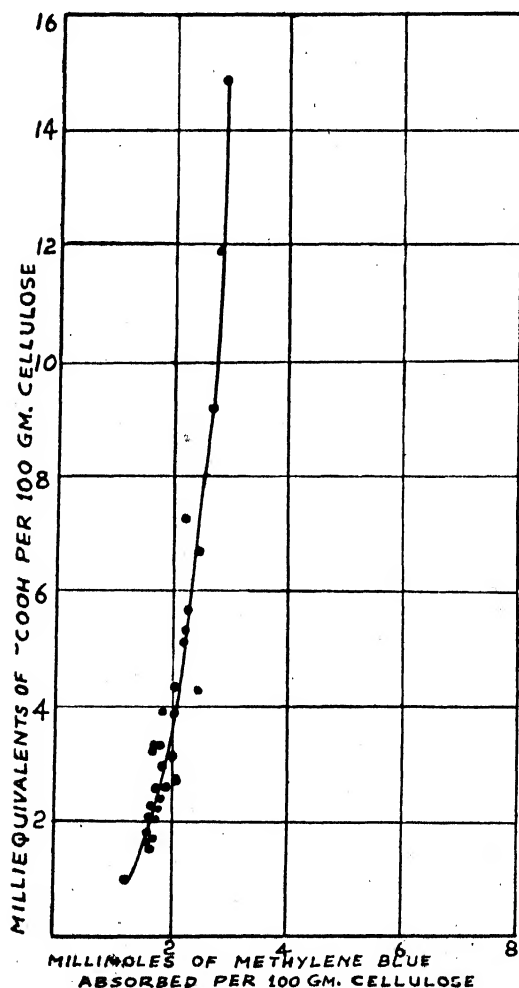


FIG. 1 — RELATION BETWEEN METHYLENE BLUE ABSORPTION AND  $-\text{COOH}$  CONTENT OF ACIDIC OXYCELLULOSE OBTAINED BY SOD. HYPOCHLORITE AND POT. PERMANGANATE TREATMENTS.

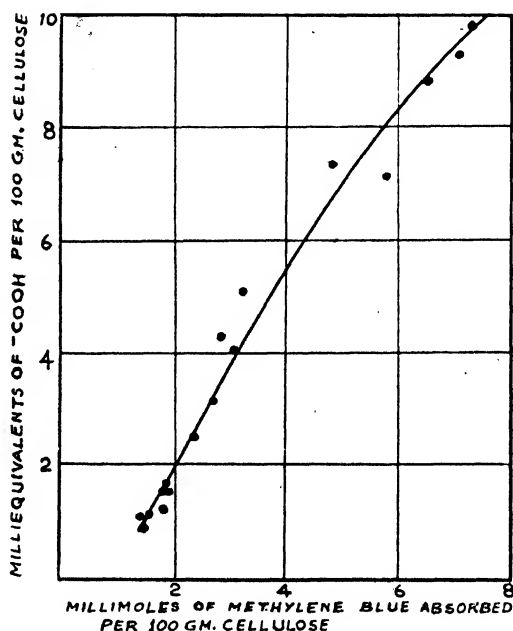


FIG. 2 — RELATION BETWEEN METHYLENE BLUE ABSORPTION AND  $-\text{COOH}$  CONTENT OF ACIDIC OXYCELLULOSES OBTAINED BY SOD. HYPOBROMITE, HYDROGEN PEROXIDE AND ACID DICHROMATE TREATMENTS.

TABLE I

STRENGTH OF METHYLENE BLUE SOLUTION	VOLUME OF METHYLENE BLUE SOLUTION C.C.	WEIGHT OF OXYCELLULOSE SAMPLE GM.	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF CELLULOSE
M/250	25	0.5	6.72
"	25	1.0	5.98
"	25	1.5	4.79
"	25	2.0	3.76
M/250	10	0.5	5.53
"	15	0.5	6.23
"	20	0.5	6.65
"	25	0.5	6.72
M/250	15	0.5	6.23
M/300	15	0.5	5.58
M/400	15	0.5	4.39
M/250	60	0.5	17.24
"	60	1.0	15.32
"	60	1.5	13.62

above this figure, this ratio increases rapidly, e.g. for a copper number of 12 this ratio has increased to 5. As suggested by Davidson<sup>7</sup>, this increase in the ratio may be attributed to the interference caused by the reducing groups during the alkali titration.

In Table III are given the results obtained with permanganate oxidized samples. The general remarks made above are equally applicable to the results recorded in this table. In view of the relatively low values of copper number of the various samples, the ratio COOH/M.B. has not deviated from unity to the same extent as was found to be the case with hypochlorite samples.

TABLE II — HYPOCHLORITE TREATMENT OF BLEACHED COTTON YARN

2 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution; temp., 30°C.

TREATMENT	OXYCELLULOSE No.	APPROXIMATE pH	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
3 gm. of available Cl. per litre, for 4 hr.	1	4	1.69	1.56	0.86	0.98
	2	5	1.68	1.74	1.24	1.04
	3	6	1.75	3.29	2.81	1.80
	4	7	2.21	7.23	7.56	3.26
	5	8	2.47	6.68	4.98	2.70
	6	9	1.80	2.20	0.80	1.22
	7	10	1.96	2.61	0.84	1.30
	8	11	2.00	2.65	0.38	1.31
3 gm. of available Cl. per litre for approximately same oxygen consumption/100 gm. of cotton	9	4	1.68	2.01	1.54	1.20
	10	5	1.72	2.20	1.72	1.28
	11	6	1.76	3.29	2.74	1.80
	12	7	1.80	3.29	2.70	1.80
	13	8	2.07	3.84	2.03	1.90
	14	9	2.43	4.30	1.76	1.70
	15	10	2.08	3.11	1.07	1.50
	16	11	2.10	2.75	0.33	1.30
For 4 hr. with 2, 3, 4, 5 gm. available Cl. per litre	17	8	2.31	5.67	4.75	2.35
	18	8	2.70	9.15	7.88	3.38
	19	8	2.83	11.90	8.74	4.20
	20	8	2.95	14.82	12.01	5.20

TABLE III — PERMANGANATE TREATMENT OF BLEACHED COTTON YARN

Nos. 21-30: 2.5 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution

Nos. 31-34: 0.5 gm. of oxycellulose in M/250 M.B. solution; temp., 30°C.

TREATMENT	OXYCELLULOSE No.	APPROXIMATE pH	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
0.04 N. pot. permanganate for 2 hr.	21	2.70	1.68	1.78	0.50	1.06
	22	4.90	1.76	2.49	0.92	1.42
	23	7.00	1.69	2.05	0.84	1.29
	24	9.15	1.82	2.40	0.78	1.32
	25	12.00	2.20	5.25	0.75	2.28
	26	2.70	1.71	2.14	0.86	1.24
0.04 N. pot. permanganate for approx. oxygen consumption equivalent to $\frac{1}{4}$ the strength of permanganate used	27	4.00	1.87	2.89	1.40	1.55
	28	7.00	1.93	3.87	1.57	2.00
	29	9.15	2.07	4.32	1.39	2.00
	30	12.00	2.20	5.07	0.87	2.53
	31	12	5.80	7.12	1.38	1.23
0.08 N. pot. permanganate for 1 hr.	32	12	6.64	8.82	1.56	1.33
2 hr.	33	12	7.11	9.32	1.67	1.31
3 hr.	34	12	7.26	9.79	1.75	1.35

**TABLE IV—SODIUM HYPOBROMITE (N/10)  
TREATMENT OF BLEACHED COTTON YARN**1.0 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution;  
temp., 80°C.; 5 hr.

OXYCEL- LULOSE No.	APPROXI- MATE pH	METHY- LENE BLUE ABSORP- TION, M. 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
35	6	2.87	4.28	1.56	1.50
36	9	4.83	7.38	2.48	1.53
37	12	2.75	3.10	0.11	1.10

**TABLE V—POT. DICHROMATE + SULPHURIC  
ACID TREATMENT OF BLEACHED  
COTTON YARN**0.5 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution;  
temp. 80°C.; 5 hr.

TREATMENT	OXYCEL- LULOSE No.	METHY- LENE BLUE ABSORP- TION, M. 100 GM. OF COTTON	CAR- BOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH B.M.
N/20 dichro- mate in N/10 sulphuric acid	38	2.30	2.43	2.65	1.0
N/20 dichro- mate in N/5 sulphuric acid	39	3.27	5.07	5.96	1.8
N/10 dichro- mate in N/10 sulphuric acid	40	3.11	4.05	5.00	1.3

**TABLE VI—TREATMENT OF BLEACHED COTTON  
YARN IN GASEOUS NITROGEN PEROXIDE**

0.5 gm. of oxycellulose in 50 c.c. of M/250 M.B. solution

TREATMENT	OXYCEL- LULOSE No.	METHYLENE BLUE ABSORPTION, M. MOLES PER 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COOH M.B.
At atmospheric pressure for 1 hr.	41	39.63	48.50	1.21
For 2 hr.	42	35.84	40.42	1.13

**TABLE VII—TREATMENT OF BLEACHED COTTON  
YARN WITH N/5 HYDROGEN PEROXIDE  
AT 80°C. FOR 5 HR.**

1.0 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution

OXYCEL- LULOSE No.	APPROXI- MATE pH	METHY- LENE BLUE ABSORP- TION, M. 100 GM. OF COTTON	CARBOXYL CONTENT M. EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
43	8	1.92	1.65	0.82	0.86
44	10	1.92	1.6	0.16	0.83
45	12	1.96	1.55	0.7	0.76

In Table IV are recorded the results obtained with hypobromite oxidized samples. It is seen that the ratio COOH/M.B. is not differing substantially since the copper number of the samples are not high.

The results obtained with dichromate oxidation are shown in Table V. In view of the fixed chromium in the samples, it is not possible to draw any conclusion from the methylene blue absorption values.

The results of nitrogen dioxide oxidized samples are given in Table VI. In spite of the very high carboxylic acid content, indicative of considerable degradation, the ratio of COOH/M.B. is very nearly one. The mechanism of this oxidation has been suggested to be a specific one, directed towards the conversion of the primary alcoholic groups in the gluco-pyranose residue to the carboxylic acid stage.

The results obtained with the hydrogen peroxide oxidized samples are summarized in Table VII. It is seen that the ratio COOH/M.B. is more or less constant and nearer to unity. The copper number of the oxidized samples are also low.

What has been said about the hydrogen peroxide samples is true of the properties of heat and light samples which have been given in Table VIII.

In Table IX are summarized the results of the calculations of aldehyde groups from copper number determinations described in Tables II to IV. The calculations were carried out on the assumption that the copper equivalent of maltose (10 cupric copper atoms to 10 cuprous copper atoms), as obtained by Richardson<sup>12</sup>, are equally applicable to cellulose. This means that the copper number values when multiplied by 1.58 give the amounts of milliequivalents of —CHO groups per 100 gm. of cellulose sample. From the calculated value of —CHO, it was possible to calculate the amounts of the carboxylic acid groups that might be formed due to Cannizaro reaction<sup>13,14</sup> ( $2\text{CHO} + \text{H}_2\text{O} \rightarrow -\text{COOH} + \text{CH}_2\text{OH}$ ) taking place in presence of the alkali. It was assumed that all the —CHO groups were affected in this manner.

## Discussion

The results described in the foregoing will have to be considered from the following two aspects: (1) the mechanism of methylene blue absorption and the factors influencing the same and (2) the utility of the alkali titration method in

TABLE VIII—EXPOSURE OF BLEACHED COTTON CLOTH TO LIGHT &amp; HEAT

0.5 gm. of oxycellulose in 15 c.c. of M/250 M.B. solution

TREATMENT	OXYCEL- LULOSE No.	METHY- LENE BLUE ABSORP- TION, M. MOLES PER 100 GM. OF COTTON	CAR- BOXYL CONTENT M.EQ. PER 100 GM. OF COTTON	COPPER No.	COOH M.B.
Exposure to light					
for 1 month	46	1.44	0.94	0.28	0.65
for 2 months	47	1.43	1.12	0.43	0.77
Heat treatment					
at 140°C. for 4 hr.	48	1.83	1.23	0.23	0.67
at 110°C. for 13 hr.	49	1.59	1.29	0.57	0.81

measuring the carboxylic acid content of oxycelluloses.

Birtwell, Clibbens and Ridge<sup>1</sup> have shown that the absorption of methylene blue by oxycelluloses or even by pure cotton is dependent to a considerable extent on the hydrogen ion concentration of the solution. In a recent publication this point has been dealt with in considerable detail by Davidson<sup>7</sup>. It is known that carefully washed hydrocellulose samples show higher methylene blue absorption than the original cotton

due to the presence of minute quantities of acid which are fixed and are thus extremely difficult to wash away. The presence of various salts also influence the methylene blue absorption, the cations of the salts competing for the carboxylic acid groups with the methylene blue. Reference to Table I clearly shows that the ratio of cotton to methylene blue solution and also the concentration of the methylene blue in the solution influence the methylene blue absorption. An increase in the concentration of methylene blue increases the absorption, while decreasing the ratio of methylene blue solution to cotton decreases the methylene blue absorption. This influence is more noticeable when the variation in the ratio of cotton to the methylene blue solution is substantial. For smaller differences in this ratio, the methylene blue absorption values are not seriously affected. The methylene blue absorption values, therefore, can only be compared with one another if they are determined under specified conditions. From Figs. 1 and 2 it appears that, provided the conditions for the determination of methylene blue absorption are kept constant, an approximate relationship between the two

TABLE IX

	OXYCELLULOSE No.	COOH— $\frac{1}{2}$ CHO	COOH M.B.	COOH— $\frac{1}{2}$ CHC M.B.	
Hypochlorite samples	1	0.88	0.98	0.49*	
	2	0.76	1.04	0.46*	
	3	1.08	1.80	0.60*	
	4	1.26	3.26	0.57*	
	5	2.74	2.70	1.10	
	6	1.57	1.22	0.87	
	7	1.95	1.30	1.00	
	8	2.38	1.31	1.16	
	9	0.80	1.20	0.47*	
	10	0.84	1.28	0.40*	
	11	1.13	1.80	0.64*	
	12	1.16	1.80	0.64*	
	13	2.24	1.90	1.08	
	14	2.91	1.70	1.12	
	15	2.27	1.50	1.09	
	16	2.49	1.30	1.07	
	17	1.91	2.35	0.80	
	18	2.93	3.38	1.0	
	19	4.99	4.20	76	
	20	5.33	5.02	80	Mean 1.10
Permanganate samples	21	1.39	06	82	
	22	1.76	42	00	
	23	1.38	29	0.81	
	24	1.58	32	0.87	
	25	4.66	28	0.03*	
	26	1.46	24	0.83	
	27	1.79	55	0.90	
	28	2.64	00	0.36	
	29	3.21	2.08	0.55	
	30	4.39	0.53	0.00*	
	31	6.06	0.23	0.04	
	32	7.49	0.33	0.14	
	33	8.00	0.31	0.12	
	34	8.41	0.34	0.15	Mean 1.11
Hypobromite samples	35	3.04	0.50	0.06	
	36	5.41	0.53	0.12	
	37	3.00	0.10	0.09	Mean

In calculating the mean values for COOH— $\frac{1}{2}$ CHO, the results marked with an asterisk are omitted, since they differ very considerably from the average value of the ratio.



measurable quantities, viz. the methylene blue absorption and carboxyl content, may be obtained. In spite of the methylene blue absorption values being carried out under specified conditions, their relationship with the carboxyl content will depend on the correctness or otherwise of the method used for the determination of carboxylic acid values. The alkali titration method of Neale and Stringfellow<sup>3</sup> for the direct estimation of carboxyl acid groups has been criticized by Davidson<sup>7</sup>, who points out that the method is only applicable with acidic oxycellulose having low reducing power, and it is pointed out that the reducing groups interfere with the determination showing higher carboxylic acid values than those actually produced by oxidation. One of the possibilities in this connection is the conversion of the aldehydic groups (through the agency of water and alkali) to the carboxylic acid and primary alcoholic stage as follows<sup>13, 14</sup>:



The possibility of this type of reaction can only exist with oxycellulose containing aldehydic groups. In the case of specific oxidation mechanisms such as those with nitrogen peroxide, this reaction will not affect the determination of carboxylic acid values by the method of Neale and Stringfellow<sup>3</sup>. If the possibility of the reaction mentioned above is considered as interfering with the determination of carboxylic acid values, the higher values of carboxylic acid groups in the case of highly reducing oxycelluloses can be accounted for. It is assumed that all the available reducing groups are affected in this manner, so that the actual amount of carboxylic acid groups estimated in any one oxycellulose sample will be higher by a quantity equal to half the total number of reducing groups present in that sample. This is a big assumption. However, it was thought worthwhile to see how far this idea could be made applicable to explain the higher values of the carboxylic acid groups obtained with reducing oxycelluloses. It is assumed that the copper number values give more or less a correct measure of the total number of the reducing groups present in the oxycellulose samples. At present there is no direct evidence to relate the copper number of oxycellulose samples to the number of reducing groups present. But if the copper equivalent of 10 copper atoms found by Richardson<sup>12</sup> for reducing mono and disaccharides such as

glucose and maltose is made applicable to cellulose, it follows that for every aldehyde group present in cellulose, 10 copper atoms will be reduced from the cupric to the cuprous stage. This will, then, enable one to calculate the amount of carboxylic acid groups actually present and the amounts formed from the reducing groups. Such calculations were carried out with the various oxycelluloses described in Tables II to IV and are summarized in Table IX. The measured values of carboxylic acid groups have been corrected by that quantity which is calculated as having been formed from the aldehydic groups. The new ratio of corrected carboxyl content to methylene blue absorption is also given in the same table. This corrected ratio is found to be much more constant and nearer to unity than the uncorrected one for all samples except for those which are produced with hypochlorite solutions having pH 7 and below. However, it is of interest to mention that these also show an approximate constant ratio of 0.5 to 0.6. It is possible that, in the case of these oxycellulose samples, all the reducing groups are not affected by alkali, so that the corrections applied on this basis are higher and thus the ratio becomes lower than that for the rest of the samples.

From what has been said above, it is clear that both the methylene blue absorption method as well as the carboxylic acid determination by alkali titration method are dependent on several factors, and, therefore; they can be considered as semi-quantitative. The method for the carboxylic acid determination, however, gives more reliable results with acidic oxycelluloses with low reducing power.

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# Letters to the Editor

## SYNERGISM OF PTERYGOSPERMIN WITH OTHER ANTI-BACTERIAL AGENTS

"BACTERIA ARE KNOWN TO BECOME resistant to anti-bacterial agents, i.e. become drug resistant at a slow or fast rate, both *in vitro* and *in vivo*. This observed resistance has become a serious limiting factor in the therapeutic efficiency of anti-bacterial agents." It has been suggested that development of drug resistance is due to the rapid multiplication of a very few highly resistant organisms present in the parent strain. Since development of resistance to one drug does not make them resistant to others, combined action of more than one anti-bacterial agent has been found to be highly effective in inhibiting the growth of micro-organisms resistant to one of the drugs. Thus, the synergistic action of penicillin with many other anti-bacterial agents, *in vitro* and *in vivo*, has been demonstrated<sup>1,6</sup>. The synergism of streptomycin with several other anti-bacterial agents including penicillin against several Gram-positive and Gram-negative microbes has been studied and recorded<sup>7</sup>. Synergism in chemotherapy is thus not only a method of suppressing the induced drug fastness developed by microbes, but also of bringing under effective control several of the already resistant ones.

The purification and anti-bacterial properties of pterygospermin, the anti-bacterial principle of the drum-stick root has already been recorded by us<sup>8,9</sup>. Since subsequent ex-

periments showed it to be highly non-toxic<sup>10</sup> also, the combined action of pterygospermin and penicillin, streptomycin or sulphapyridine against different microbes was studied. This is a preliminary communication in which we record a summary of the results of *in vitro* studies on the bacteriostatic action of pterygospermin in the presence and absence of small amounts of penicillin, streptomycin and sulphapyridine.

Crystalline sodium penicillin G, a standard sample of streptomycin (Merck & Co.), crystalline sulphapyridine and pterygospermin prepared by the method described by us<sup>9</sup> were used for these studies. The anti-bacterial activity of individual drugs and of mixtures of known composition were found out by the serial dilution method. Synergistic effects obtained for *S. aureus* and *E. coli* are presented in this communication.

The results clearly indicate that in the presence of non-inhibitory concentrations of penicillin and streptomycin, the anti-bacterial properties of pterygospermin are increased considerably. The minimum inhibitory concentrations of penicillin and streptomycin are also much lowered in presence of small quantities of pterygospermin. In view of the non-toxic nature of pterygospermin, the above findings are likely to assume great practical importance. The ability of pterygospermin to inhibit the growth of penicillin and streptomycin-resistant bacteria, the inability of -SH compounds to inactivate it, all seem to indicate that the mode of action of pterygospermin

TABLE I — SYNERGISM OF PTERYGOSPERMIN WITH STREPTOMYCIN

ORGANISM	GROWTH IN									CONTROL	
	PTERYGOSPERMIN ALONE IN PTERYGOSPERMIN UNITS PER C.C.			STREPTOMYCIN ALONE IN STREPTOMYCIN UNITS PER C.C.			MIXTURES				
							Streptomycin units per c.c. ...	Pterygospermin units per c.c.			
	1.5	1.0	0.75	2.0	1.0	0.5					
<i>S. aureus</i>	...	...	++	...	...	++	{ 0.5 0.25 0.10				+++
									++	++	
	6	4	3.5	2.0	1.0	0.5		3	1.5	1.0	
<i>E. coli</i>	...	...	++	...	...	++	{ 0.5 0.25 0.10				+++

TABLE II — SYNERGISM OF PTERYGOSPERMIN WITH PENICILLIN

GROWTH IN												
ORGANISM	PTERYGOSPERMIN ALONE, UNITS PER C.C.			PENICILLIN ALONE, UNITS PER C.C.				Penicillin units per c.c.	MIXTURES Pterygospermin units per c.c.			CONTROL PER C.C.
	1.5	1.0	0.75	0.85	0.03	0.02	0.01		0.75	0.5	0.25	
<i>S. aureus</i>	...	...	++	...	+	+	++	{ 0.03 0.02 0.01	...	...	...	+++
	6.0	4.0	3.5	5.0	4.0	3.0	++		++	++		
	...	...	++	...	++	++	1.85		0.75	...		
<i>E. coli</i>	...	...	++	...	++	++	{ 5.0 4.0 3.0 2.0	...	...	...	+++	
	...	...	++	...	++	++		...	...	...		
	...	...	++	...	++	++		++	++	++		

TABLE III — COMBINED ANTI-BACTERIAL ACTION OF PTERYGOSPERMIN & SULPHAPYRIDINE AGAINST *S. AUREUS*

PTERYGOSPERMIN UNITS PER C.C.	SULPHAPYRIDINE DILUTION X 10	GROWTH
Nil	Nil	+++
Nil	0.2	+
0.75	Nil	+
0.75	0.2	+
0.75	0.1	+
0.75	0.02	++
0.375	0.2	++
0.375	0.1	++
0.375	0.02	++

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is widely different from either that of penicillin or streptomycin.

Synergic effects observed in mixtures of sulphapyridine and pterygospermin are of a very low degree and may not be of much practical utility. Bacteria resistant to pterygospermin appear to be not affected by sulphapyridine as well. It is not, however, possible to infer anything regarding the similarity or otherwise of the mode of action of the two compounds. Detailed investigations on the synergic action of pterygospermin with other anti-bacterial agents, against other microbes, and the efficiency of the synergic mixtures *in vivo* are in progress.

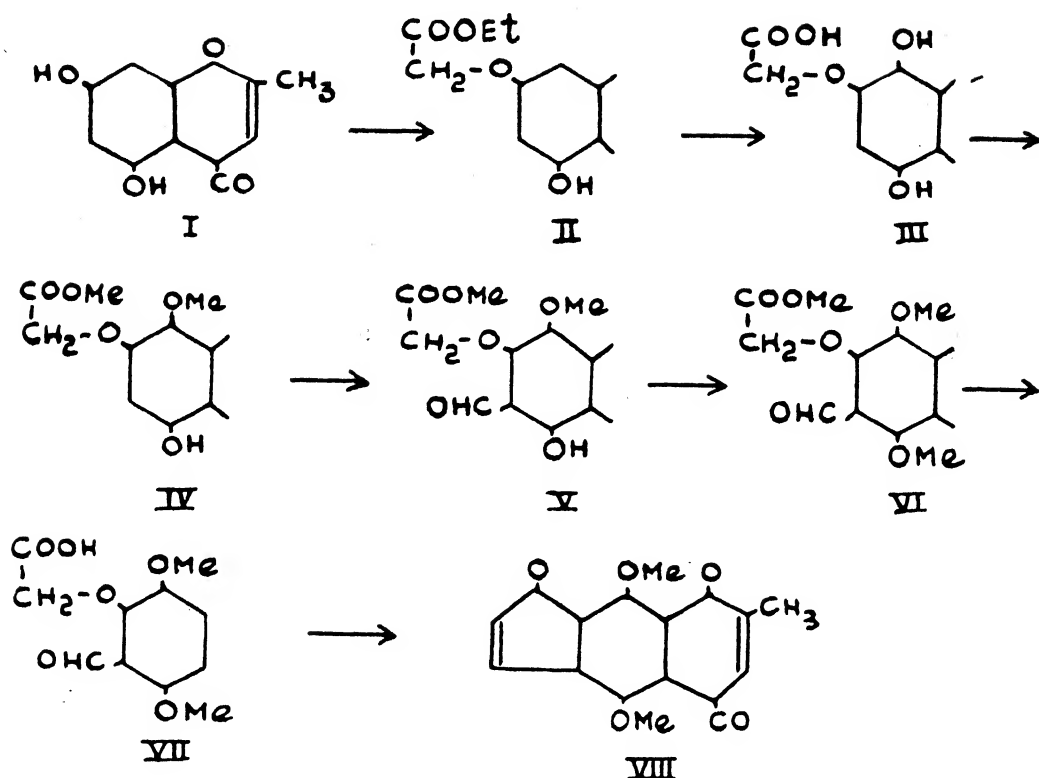
Our thanks are due to Dr. K. M. Pandalai, Dr. N. N. De and Prof. V. Subrahmanyam for their interest and helpful suggestions, to Merck & Co., Rahway, N.J. for the supply of streptomycin used, and to the Council of Scientific & Industrial Research for financial assistance.

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September 13, 1948

## A SYNTHESIS OF KELLIN

AMONG THE NATURALLY OCCURRING compounds having a fused chromone and furan ring systems, kellin has recently assumed great importance because of its use in the treatment not only of leucoderma but also of spasmodic conditions such as asthma and intestinal colic and of certain diseased conditions of the heart. Its constitution was established by Spath and Gruber<sup>1</sup> but its synthesis has not so far been published. Based on our work of the past two years on nuclear oxidation in flavones and related compounds, it has been possible for us to effect this synthesis. The starting material is 5:7-dihydroxy-2-methyl chromone (I) whose preparation has been made in improved yields. It is next condensed with 1 mole of bromacetic ester in acetone solution in the presence of anhydrous potassium carbonate, the product being (II). Nuclear oxidation of it with alkaline persulphate gives rise to the quinol (III) which is partially methylated to form (IV). Condensation with hexamine in glacial acetic acid solution yields the aldehyde (V) which is subsequently subjected to complete methylation (VI),



and gentle hydrolysis with dilute alkali, yielding the aldehydo-acid (VII). Boiling with sodium acetate and acetic anhydride finally produces synthetic kellin (VIII) which is found to be identical with natural kellin from *Ammi visnaga*.

Our thanks are due to Professors A. Schonberg and R. B. Fahmy of Cairo for the supply of samples of natural kellin.

V. V. S. MURTI  
T. R. SESHADRI

Department of Chemistry  
Andhra University, Waltair  
April 25, 1949

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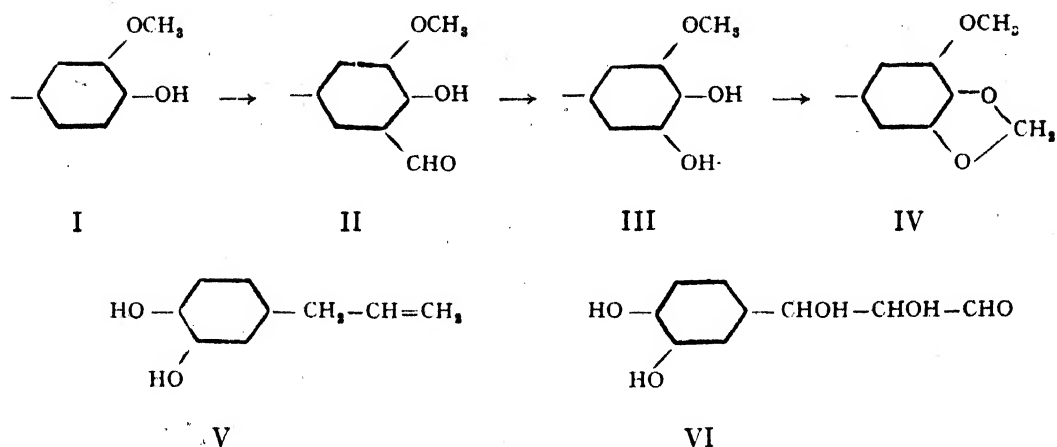
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#### A SYNTHESIS OF MYRISTICIN & ELEMICIN

IN THE STUDY OF NUCLEAR OXIDATION IN the flavone series, it has been shown that *para*-oxidation takes place readily with alkaline persulphate and it could be used

in a number of ways<sup>1</sup>. On the other hand, *ortho*-oxidation does not proceed satisfactorily by this method. A two-stage process formulated below (I to III) was, therefore, adopted and its suitability proved by the synthesis of myricetin<sup>2</sup> and robinetin<sup>3</sup> involving *ortho*-oxidation in the side phenyl nucleus. One of the synthetic advantages of this method is in the preparation of methylene ethers (IV) and as a typical example the synthesis of kanugin<sup>3</sup> has been carried out.

This work has now been extended to the components of essential oils which are classified as allyl benzene derivatives. Robinson<sup>4</sup> has drawn attention to the biogenetic similarity between the nine carbon system present in these (V) and in the non-phloroglucinol part of the anthocyanins and anthoxanthins arising from component B of the biogenetic precursor (VI). The derivatives of allyl pyrogallol like myristicin (X) and elemicin (XI) may, therefore, be considered to arise from those of allyl catechol by nuclear oxidation. In support of this view, the two-stage *ortho*-oxidation process has now been carried out with



eugenol (VII), probably the most important member of the allyl-catechol type. By the action of hexamine it readily forms aldehydo-eugenol (VIII) which undergoes oxidation with hydrogen peroxide to hydroxy-eugenol (IX). Methylenation of (IX) with methylene dibromide yields myristicin (X) and methylation with dimethyl and

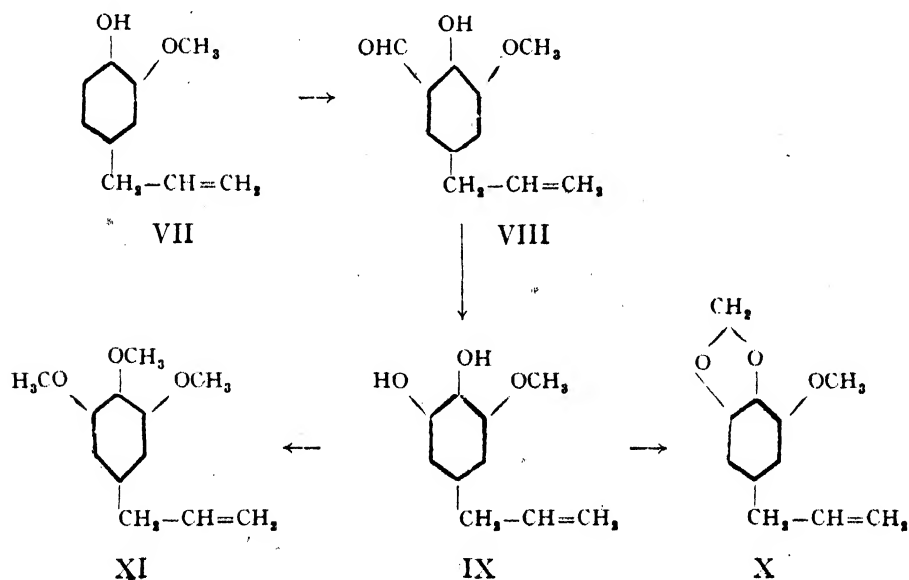
elemicin isomerized to *iso*-elemicin and oxidized to trimethyl gallic acid.

K. V. RAO

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*Department of Chemistry*  
*Andhra University, Waltair*  
*April 4, 1949*



sulphate yields elemicin (XI). The products have been characterized by a study of the physical properties (boiling point and refractive index), by analysis and also by conversion into derivatives. Myristicin has been converted into dibromomyristicin dibromide

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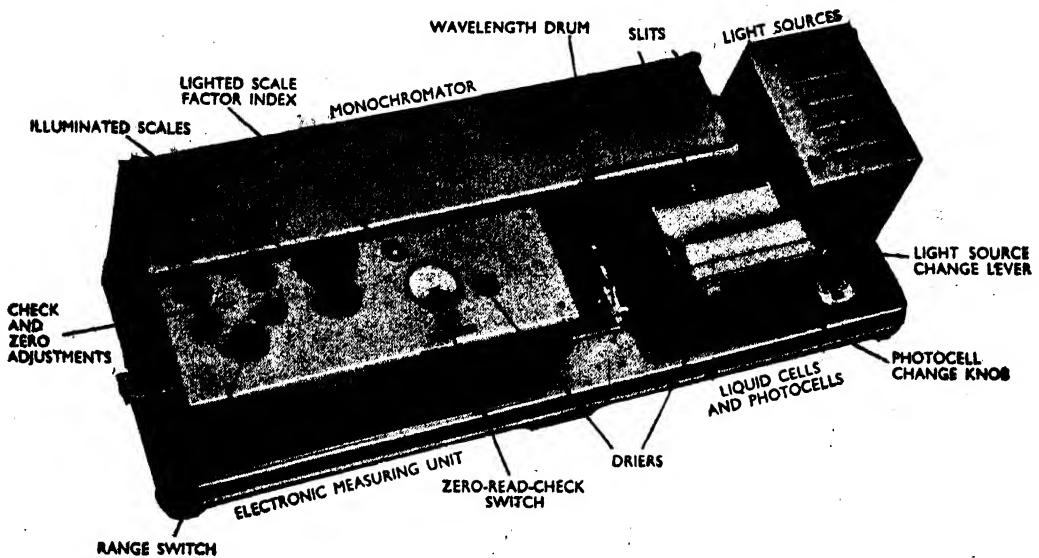
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# Chemical Examination of *Tinospora cordifolia*, Miers

AKHLAQ-UR-REHAMAN KIDWAI, KAILASH CHANDER SALOOJA, VISHWA NATH SHARMA & SALIMUZZAMAN SIDDIQUI

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Delhi

**T**INOSPORA *cordifolia* Miers (Hindi — *Giloe, Gulanch*) is a well-known indigenous drug commonly used as a febrifuge, an antiperiodic and as a general tonic. The fresh plant material is extremely bitter in taste.

Pluckiger<sup>1</sup> reported that the therapeutic properties of the drug as well as its bitterness are due to the presence of a glucoside and traces of berberine. Kirtikar<sup>2</sup> also ascribes the medicinal properties to the presence of berberine. Pendse and Dutt<sup>3</sup> reported the presence of a waxy substance and an alkaloidal constituent different from berberine. They further stated that the bitter constituent was a semi-solid product which could not be crystallized. Subba Jois<sup>4</sup> isolated from the material three crystalline substances melting at 75°-77°C., 83°-84°C. and 181°C. respectively. Out of these, the substance melting at 181°C. was assigned the molecular formula  $C_{16}H_{18}O_5$ , while the product melting at 83°-84°C. was noted to be a monohydroxy alcohol of the fatty series. Bhide, Phalnikar and Paranjpe<sup>5</sup> isolated two bitter substances: (A)  $C_{22}H_{34}O_{10} \cdot 5H_2O$ , m.p., 226°-228°C.; and (B) m.p., 186°-188°C., and a neutral fatty alcohol,  $C_{28}H_{58}O$ , m.p. 82°-83°C.

In view of the great importance attached to *Giloe* in the indigenous systems of medicine and the varied claims put forward by different authors in regard to its active constituents, a systematic chemical examination of the plant was undertaken. As a result of these investigations, the following crystalline substances have been isolated and characterized:

1. Giloin,  $C_{23}H_{32}O_{10} \cdot 5\frac{1}{2}H_2O$ ; glucoside; m.p., 226°-228°C.;  $[\alpha]_D^{25} = +69.5^\circ$  (after drying to constant weight *in vacuo* over  $P_2O_5$ ) in 1 per cent alcoholic solution; yield 0.2 per cent on the weight of fresh bark-free stem; bitter in a dilution of 1 in 10,000.

2. Giloinin,  $C_{17}H_{18}O_5$ ; non-glucosidic; bitter; m.p., 210°-212°C.;  $[\alpha]_D^{25} = +40^\circ$  in 1 per cent acetone solution; yield, 0.001 per cent on the weight of fresh bark-free stem; bitter in a dilution of 1 in 1,000.

3. Gilo-sterol,  $C_{28}H_{48}O$ ; m.p., 192°-193°C.;  $[\alpha]_D^{25} = +92^\circ$  in alcoholic solution.

The method worked out for the isolation of the bitter crystalline constituents was based on the extraction of the bark-free, crushed stems with water in the hot or alcohol at the ordinary temperature, concentration of the combined extracts under reduced pressure, and separation of the different fractions from the aqueous concentrate with the help of miscible and non-miscible solvents and solvent mixtures, without the use of any chemical reagents. The gilo-sterol was isolated from the unsaponifiable fraction of fatty matter through crystallization from organic solvents. The mixture of giloin and giloinin which initially formed the crude crystallizate obtained on dissolving the ether-insoluble fraction in dilute alcohol and concentration of the solution on the water bath, yielded the individual crystalline substances after a long process of repeated fractional crystallization. There was considerable wastage involved in this process of separation but



it yielded pure products which showed no increase in their melting points on subsequent crystallization.

Giloin is sparingly soluble in ether, petrol ether and benzene, soluble in water and ethyl acetate in the hot and fairly soluble in alcohol, methanol and acetone. It does not contain any methoxy group. It could not be catalytically reduced with platinum black and does not readily absorb bromine in the cold, which would indicate the absence of ethylenic linkages. Giloin is fairly stable to acid hydrolysis but is readily hydrolysed on warming with dilute alkali for a few minutes. Due to its susceptibility to alkaline hydrolysis, it could not be benzoylated by Schotten-Baumann's method, nor methylated with dimethyl-sulphate and alkali. The sugar component of the glucoside was identified as glucose through its osazone. The aglucone could not be isolated, but from the products of acidic hydrolysis, crystalline barium and silver salts of an organic acid have been obtained which are under investigation. Giloin appears to be an  $\alpha$ -glucoside as it is not hydrolysed by emulsion. It does not give tests for aldehydes and ketones but reduces Fehling solution due apparently to its initial hydrolysis in alkaline medium. The melting point of giloin would indicate that it is identical with the crystalline product (A) isolated by Bhide and coworkers. In so far, however, as Bhide *et al.*'s product shows dextro rotation of  $48^\circ$  and analyses for  $C_{22}H_{34}O_{10}$  with C, 57.6; H, 7.4 per cent as against giloin,  $C_{23}H_{32}O_{10}$ ,  $[\alpha]_D^{25} = +69.5^\circ$  with C, 59.0; H, 6.8 per cent, the latter appears to be definitely different from the former. This conclusion gets further support from the fact that in contrast to substance (A), giloin has been established as a glucoside. Bhide *et al.*'s substance (A) failed to reduce Fehling solution after attempted hydrolysis and it was considered by them to be non-glucosidic in character.

Giloinin does not contain any methoxy group and fails to give an acetyl derivative or a phenyl hydrazone. It is recovered unchanged on heating with dilute acids on the water bath, but on treatment with dilute alkali, it is degraded to water-soluble products from which no crystalline substance could be isolated. It does not give any colouration with ferric chloride and does not reduce Fehling solution.

Giloinin is quite distinct from substance (B), reported by Bhide, Phalnikar and

Paranjpe, which melts  $24^\circ$  lower. As no molecular formula has been assigned to their product, no suggestion about its possible relationship to giloinin can be made at this stage.

Comparing the molecular formula of giloin,  $C_{23}H_{32}O_{10}$ , with that of giloinin,  $C_{17}H_{18}O_5$ , it was expected that giloinin might prove to be the aglucone of the glucoside. As already stated, however, it has not so far been possible to isolate giloinin or any other aglucone from the hydrolysate of giloin. Further work on the elucidation of this point is in progress.

From the fatty portion, a number of lower melting crystalline substances corresponding to the waxy products reported by the previous authors were obtained, but these were not pursued further. Gilo-sterol, which has not so far been reported by any of the previous authors, was isolated from the unsaponifiable fraction of the fatty matter. It conforms to the formula  $C_{28}H_{48}O$  and showed  $[\alpha]_D^{25} = +92^\circ$  in alcoholic solution. It gave all the colour reactions of sterols and formed a crystalline acetyl derivative and the digitonide.

### Experimental

25 lb. of the fresh stem of *Tinospora cordifolia*, stripped of the green bark and then crushed, was extracted 4 times with water in the hot at  $80^\circ$ - $100^\circ$ C., till the residue was no longer bitter to taste. The aqueous extracts were concentrated under reduced pressure to a thick syrupy liquid (5 kg.) which was treated with 5 gallons of rectified spirit. The alcoholic solution was filtered off from the insoluble matter and concentrated to a small volume *in vacuo*. After a repetition of this process with the concentrate for eliminating most of the starchy matter, the semi-solid residue was successively extracted with ether and benzene. The ether-benzene insoluble portion yielded, through its dilute alcoholic solution, a mixture of crude giloin and giloinin (23 gm.) which were separated through a process of purification and repeated fractional crystallization from alcohol as described later. The combined ether-benzene extracts gave a further small quantity of crystallize consisting mainly of giloinin, which was separated off. The filtrate was freed of the solvent and the residue consisting of the total fatty constituents was saponified with alcoholic potash. The unsaponifiable matter was separated from the acids in the usual manner, when,

on repeated crystallization through acetone, it finally yielded gilo-sterol, m.p., 192°C.

In a separate working, 25 lb. of the fresh, crushed material were repeatedly percolated with alcohol at the ordinary temperature. The extracts were concentrated under reduced pressure and the concentrate digested with benzene. The benzene layer on removal of the solvent gave nearly 50 gm. of the fatty constituents. The benzene-insoluble residue was dissolved in alcohol and the solution was filtered, charcoaled, concentrated *in vacuo* and kept in the cold, when colourless needles melting at 215°-220°C. separated out. The mother liquors, on further concentration, gave a further crop of crystals, ca. 1 gm., consisting mainly of giloinin making up a total yield of 22.5 gm. of the crude crystallizate consisting of giloin and giloinin. The final mother liquors which were not appreciably bitter were rejected.

*Giloin* — The crude crystallizates consisting of a mixture of giloin and giloinin were dissolved in alcohol, and the solution treated with ether and a small quantity of petrol ether which removed coloured impurities. The clarified solution was concentrated and the alcoholic concentrate diluted with water to turbidity, when, on keeping in the cold, straw-coloured needles of giloin melting at 220°-222°C. separated out, which were filtered, washed with dilute alcohol and dried on a porous plate. After repeated crystallizations from dilute alcohol, dilute acetone and moist ethyl acetate, giloin was finally obtained in the form of colourless, silky needles melting at 226°-228°C. There was no rise in the melting point on further crystallization from any of the solvents. On drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, giloin crystallized from dilute alcohol, dilute acetone and moist ethyl acetate showed a loss of 17.9, 17.2 and 17.7 per cent respectively;  $C_{23}H_{32}O_{10} \cdot 5\frac{1}{2}H_2O$  requires  $H_2O$ , 17.4 per cent. Found after drying: C, 59.0, 59.3, 59.1, 59.4 per cent, mean, 59.2 per cent; H, 7.0, 7.0, 6.9, 6.9 per cent, mean 6.9 per cent; M.W. (cryoscopic in phenol), 496;  $O-CH_3$  (after Zeisel), nil;  $C_{23}H_{32}O_{10}$  requires C, 59.0; H, 6.8 per cent; M.W., 468. An air-dried sample in 1 per cent alcoholic solution showed  $[\alpha]_D^{25} = +59^\circ$ , while the dried product showed  $[\alpha]_D^{25} = +69.5^\circ$ .

Giloin is nearly insoluble in petrol ether, sparingly soluble in ether or benzene, fairly soluble in alcohol, acetone, methanol, moist

ethyl acetate and water in the hot. Giloin does not show any colouration with ferric chloride. It reduces Fehling solution but fails to react with phenyl hydrazine or semicarbazide. It could be recovered unchanged on evaporation of its solution in glacial acetic acid or liquor ammonia on the water bath. It dissolves in cold, concentrated sulphuric acid to a dark-red solution which, on dilution with cold water, gives a cream-coloured precipitate from which some unchanged giloin could be recovered. It dissolves in dilute alkali or saturated baryta on slight warming, but does not separate out on cooling or acidification of the solution due apparently to hydrolysis. It is also hydrolysed when heated with 10 per cent hydrochloric acid on the water bath or on heating with liquor ammonia in a sealed tube at 100°C. The products of hydrolysis in all these cases were water soluble and no crystalline product could be isolated from the reaction mixture.

*Hydrolysis of Giloin with Dilute Sulphuric Acid* — Giloin (2 gm.) was heated with 200 c.c. of 7.5 per cent sulphuric acid on a water bath for 3 hours. The resultant yellow-coloured solution which showed a greenish fluorescence was shaken out with ethyl acetate. The ethyl acetate extract was washed with water, dried over anhydrous sodium sulphate, filtered and freed of the solvent, when a reddish treacly residue which was insoluble in petrol ether and ether but soluble in other organic solvents was obtained. It was dissolved in ethyl acetate and purified through the addition of ether and a little petroleum ether. The resulting yellowish solution yielded a small quantity of a crystalline deposit (0.07 gm.) melting indefinitely between 245°C. and 252°C. which is under investigation.

The acidic layer was treated with barium carbonate till the solution was neutral, and the filtrate separated from barium sulphate was evaporated to dryness on a water bath. The residue was repeatedly washed with methanol, when a crystalline, colourless product was left behind which gave 53.7 per cent barium. An aqueous solution of the barium salt on treatment with silver nitrate yielded the corresponding silver salt in the form of colourless shining needles, which after drying to constant weight *in vacuo* over phosphorus pentoxide at 100°C. gave C, 14.3, 14.6 per cent; H, 1.9, 1.7 per cent; Ag, 62.0, 64.6 per cent (as AgI).

The methanol solution was freed of the solvent and the residue was taken up in alcohol and purified through ether and a small quantity of petrol ether. The clear solution was concentrated and then refluxed with phenyl hydrazine and 50 per cent acetic acid on the water bath for one and a half hours. The osazone of the sugar component of the glucoside separated out in yellow needles which were filtered, washed with water and dried on the plate. After crystallization from alcohol, it melted at 202°C. and did not show any depression in melting point on admixture with pure glucosazone.

**Isolation & Characterization of Giloinin** — The combined ether-benzene extracts of the alcoholic extractive of the drug gave, on keeping, a small fraction of crude giloinin melting at 190°-200°C. After repeated crystallizations from a mixture of alcohol and acetone, giloinin was obtained in the form of colourless, silky needles melting at 210°-211°C. Another alcohol-insoluble crystalline substance was also obtained during the process of purification of giloinin which melted at 286°C., but its quantity was too small for a detailed investigation. A further small quantity of giloinin could also be obtained in the course of the fractional crystallization of the crude crystallizate obtained from the ether-benzene insoluble portion of the alcoholic extractive which mainly consisted of giloin; total yield, 0.001 per cent on the weight of fresh, bark-free stem.

Giloinin is sparingly soluble in petrol ether, ether and benzene, fairly soluble in alcohol, and methanol in the hot, more readily in acetone and chloroform. From alcohol, it crystallizes in colourless, short, prismatic rods. In 1 per cent acetone solution, it showed  $[\alpha]_D^{25} = +40^\circ$ . It does not contain any methoxyl group, and does not reduce Fehling solution. The air-dried substance does not suffer any appreciable loss in weight on drying *in vacuo* at 100°C. over phosphorus pentoxide. Found after drying: C, 66.8, 67.0 per cent; H, 6.3, 6.15 per cent; M.W. (cryoscopic in phenol), 331;  $C_{17}H_{18}O_8$  requires: C, 67.5 per cent; H, 6.0 per cent and M.W., 302.

Giloinin dissolves in dilute alkali on slight warming and is not precipitated out on acidification. On treatment with acetic anhydride and fused sodium acetate, also after heating with 5 per cent sulphuric acid, the substance is recovered unchanged. It does

not give any colouration with ferric chloride in alcoholic solution.

**Isolation & Characterization of Gilo-sterol** — After the separation of giloinin from the ether-benzene soluble product, the fatty residue was saponified with 10 per cent alcoholic potash on a water bath. The dark-red hydrolysate was diluted with water and extracted with ether. The ethereal extract was washed with water, dried over anhydrous sodium sulphate and freed of the solvent by warming on a water bath. The residue (15.8 gm.) was kept in the cold in acetone solution, when clusters of colourless needles melting within a range of 151°-158°C. separated out; yield 5.5 gm. On repeated alternate crystallizations from petrol ether and acetone, the crystallizate finally yielded pure gilo-sterol melting at 190°-192°C. A number of lower melting fractions melting between 157°C. and 182°C. were also obtained in the course of the fractional crystallization, but their quantities were too small for detailed study.

Gilo-sterol gives the Liebermann-Burchard reaction and forms a digitonide. It is fairly soluble in petrol ether, alcohol methanol and acetone in the hot, readily soluble in ether and chloroform. In alcoholic solution, it gave  $[\alpha]_D^{25} = +92.8^\circ$ . Found after drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide: C, 83.7 per cent; H, 11.9 per cent; M.W. (after Rast), 399.  $C_{28}H_{48}O$  requires: C, 84.0 per cent, H, 12.0 per cent and M.W., 400. The acetyl derivative prepared in the usual manner melted at 234°C. with previous shrinking at 220°C.

#### Acknowledgement

The authors take this opportunity to thank Drs. Weiler and Strauss and Mr. R. C. Tewari for some of the analytical data embodied in the paper. One of the authors (A. K.) is also deeply indebted to Messrs Cipla Ltd., Bombay, for the facilities provided to him in the early stages of these investigations.

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# A Method for the Production of Carotene Concentrate from Water Hyacinth (*Eichhronia crassipes*, Solms)

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SEVERAL workers have pointed out the possibility of production of carotene from water hyacinth (*Eichhronia crassipes*, Solms) which is regarded as a pest in Bengal. It grows luxuriantly in ponds and, according to official reports, it has been spreading over an area of 4,269 sq. miles in the lower districts of Bengal. It has been calculated that about 107 million tons of green water hyacinth can be easily had in Bengal. No process or method for the production of carotene from water hyacinth appears to have been recorded so far. A method developed for the production of carotene concentrate on a laboratory scale has been worked out, and described here.

The method consists of the following stages: (1) dehydration of the leaves; (2) extraction of the total pigment by an organic solvent; (3) purification of the extract by chromatographic separation; (4) concentration and recovery of the solvent; and (5) storage.

## Experimental

Water hyacinth leaves were collected at Singur, 25 miles away from Calcutta, and were immediately treated as follows:

**Dehydration**—Lots of 1,200 gm. of fresh leaves were dehydrated by several procedures. In one set of experiments the untreated leaves were placed inside an electric air oven at 60°C. In another set, leaves were chopped and blanched (immersion in boiling water in an aluminium vessel for 3 min.) and dried in an air oven at 60°±5°C. Fig. 1 represents graphically the course of dehydration in the samples treated. The carotene contents of samples treated by the 2 methods are recorded in Table I. The untreated leaves on dehydration show considerable discolouration, while the blanched samples retain much of the greenness.

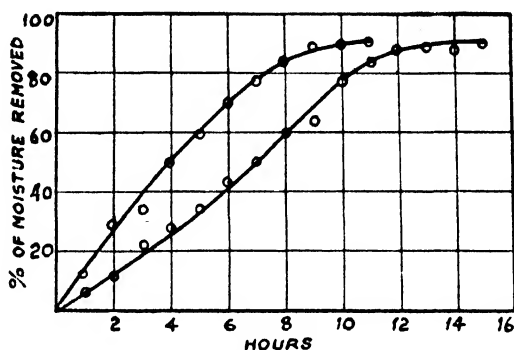


FIG. 1

TABLE I—CAROTENE CONTENT (ON DRY BASIS)  
(mg. per 100 gm.)

UNTREATED	BLANCHED	DIFFERENCE
16.86	28.1	40.1
19.40	35.3	45.0
20.93	34.2	41.7
18.18	31.8	42.8
21.21	36.2	41.4

Blanching hastens dehydration and raises the yield of carotene by about 42 per cent.

**Extraction**—Extraction was conducted in (1) a glass Soxhlet (2 litres); (2) copper Soxhlet (3 litres); and (3) long-necked, round-bottomed flask (2 litres), fitted with a reflux condenser for direct boiling on water bath. Petroleum ether (b.p., 40°-60°C.) was used for Soxhlet extraction and high-boiling petroleum ether (b.p., 80°-100°C.) was used for direct heating. The yields of carotene are shown in Table II.

It is seen that a maximum yield of 85.3 per cent is attainable when extractions are made by direct heating over a water bath employing high-boiling petroleum ether.

**Purification of the Extracts**—As phasic separation is unsatisfactory and expensive for adoption on a commercial scale, chromatographic separation was adopted for the purification of the extract. A mixed adsorbent consisting of soda ash and light

**TABLE II — 100 GM. OF DRIED LEAVES EXTRACTED FOR 6 HR.**

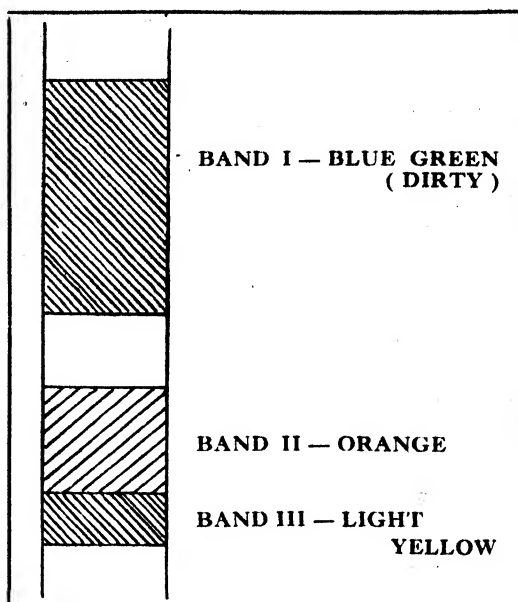
MOISTURE %	CAROTENE MG.	PETROLEUM ETHER 40°-60°C.				PETROLEUM ETHER 80°-100°C.			
		1		2		3			
		Yield	Recovery %	Yield	Recovery %	Yield	Recovery %		
84.7	35.1	21.4	61.0	18.6	53.0	29.7	84.6		
86.1	28.7	17.04	59.0	15.9	55.0	24.6	85.7		
88.3	32.7	18.30	56.1	17.1	52.2	28.4	86.0		
85.7	39.7	23.02	59.0	18.7	47.0	34.4	86.6		
87.6	37.1	22.33	60.2	18.97	51.1	30.7	82.7		
AVERAGE									
86.4	34.7	22.24	59.2	17.6	51.6	29.6	85.3		

magnesia (*Darlington Brand*) was found to be satisfactory for chromatographic separation. Soda ash is cheap and selective in adsorption. Of the adsorbed materials, the carotenoid pigments, being the least adsorbed constituents of the mixture, passed through while the contaminants, chlorophylls and allied chromogens remained bound to the adsorbent. There was little decomposition of the pigments during the chromatographic analysis. A battery of 3 or more large adsorption tubes, 2"×10", was employed. Each tube was uniformly packed with an intimate mixture of soda ash and magnesia (6:1). The tubes were fitted into rubber stoppers fitted to suction flasks. Petroleum ether (40°-60°C.) was forced through the tubes until the columns were covered with the solvent. The extract was then poured into the tubes and washed with fresh petroleum ether. The process was continued until the carotenoid column (least adsorbed) was washed into the filtering flasks, the washing being deemed complete when the washings began to show the brownish colour of chlorophylls. The principal bands obtained during the chromatographic separation are shown diagrammatically in Fig. 2.

The orange (2) and light-yellow (3) bands were taken together as total carotenes.

The carotene concentrate prepared as above contained a mixture of isomeric forms of carotenes.

**Analytical Methods** — For the analysis of fresh leaves, the method of Moore<sup>4</sup> was adopted with suitable modification. 4.5 gm. of leaves were minced and refluxed with 50 c.c. of 95 per cent alcohol for 30 min. The extract was filtered into a separator, and the residue further refluxed with 25 c.c. alcohol, filtered, and the filtrate added to the former extract. A mixture containing 90 per cent methanol and petroleum ether

**FIG. 2 — CHROMATOGRAPHIC ADSORPTION OF EXTRACT.**

(b.p., 40°-60°C.) was used for washing the residue. The residue was macerated in a mortar and re-extracted with further 25 c.c. portions of petroleum ether containing a little methanol. The extraction was continued till the residue was almost colourless. 50 c.c. of water and 2-3 gm. of sodium chloride were then added to the separator containing the combined extracts, shaken, and the two phases were allowed to separate. The alcohol water layer was transferred to a second separator and re-extracted with 25-30 c.c. portions of petroleum ether. The partitioning was repeated 2 or 3 times. The petroleum ether solution was washed with water and dehydrated with anhydrous sodium sulphate. The solution was then chromatographed on a column of 2"×10", made of the mixed adsorbent described above. The carotene in the solution was estimated using the Evelyn photoelectric colorimeter with filter No. 420, with the aid of a reference curve showing the relationship between colorimeter readings and the concentration of carotene in a standard solution of  $\beta$ -carotene in petroleum ether.

The dried leaves were treated according to the method (E) described by Nelson<sup>5</sup> for dried grass, with the modification that the adsorbent used was a mixture of soda ash and magnesia (6:1). 2-2.5 gm. of weighed samples were extracted in a Kjeldahl flask with 50-60 c.c. of B.D.H. (Analar)

petroleum ether (b.p., 80°-100°C.) for 2-2.5 hr. and the solution decanted. The residue was further extracted for half an hour with 25 c.c. of petroleum ether and filtered. The combined extracts were purified by chromatographic separation and the carotene concentration estimated as above.

**Yield of Carotene** — 3 kg. of fresh, chopped leaves were blanched in boiling water for 3 min. and dried in an electric air oven at 60°C. The dried leaves were pulverized and extracted in a series of 3 long-necked flasks with 1 litre of petroleum ether (b.p., 80°-100°C.) for 5 hr. The extract was decanted, and the residue treated with further 100 c.c. of the solvent for half an hour. The combined extract was concentrated to a fourth of its volume and subjected to chromatographic separator in a battery of 3 adsorption tubes (2" × 10") using the mixed adsorbent. The carotenoid pigments were washed with petroleum ether (b.p., 60°-70°C.), collected in a suction flask, and the solvent recovered by distillation under reduced pressure. The residual petroleum ether was removed by a draught of carbon dioxide. The final product was kept in sealed bulbs wrapped in black paper. The results obtained are given in Table III.

TABLE III  
(3 kg. fresh leaves extracted)

MOISTURE %	CAROTENE CONTENT (FRESH BASIS) MG.	YIELD (FRESH BASIS) MG.	RECOVERY
87.1	168.3	113.1	84.0
84.9	163.8	138.7	84.6
85.4	156.3	134.6	86.1
86.0	172.7	146.7	87.9

**Biological Assay** — A group of 6 normal young rats (1 male and 5 females) were

maintained on a vitamin A-free diet for about 6 weeks to reduce the body store of the vitamin. The animals were fed on a daily dose of 50 γ of the test product dissolved in 0.1 c.c. of olive oil for a period of 4 weeks. They were weighed at weekly intervals. The average weights of the test animals at the end of the successive weeks are shown in Fig. 3, along with those of rats fed on a normal diet during the same period. There is a graded gain in the weights of test animals after the intake of the product at the end of the depletion period.

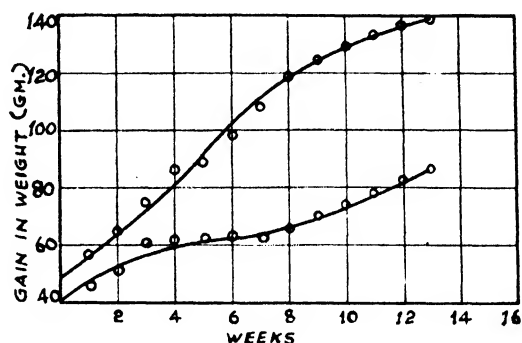


FIG. 3

#### Acknowledgement

The authors' thanks are due to Shree G. Karmarkar for valuable assistance in the spectro-photometric estimation of carotene.

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## Activation of Gangetic Silt

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**I**NVESTIGATIONS on the bleaching properties of Fuller's earth reveal that this is due either to adsorption or absorption phenomenon or to both. Other clays also possess bleaching properties though

not to the same extent as Fuller's earth. The bleaching property of such clays can be improved by activation, i.e. by submitting these to certain pre-treatments. Even Fuller's earth has been found to

acquire better adsorptive properties on pre-treatment.

Activated earth and clays find use in the refining of animal, vegetable and mineral oils.

Deposits of Fuller's earth are found in Jodhpur, Jaisalmeer, Jubbulpore and other places in India. Kaolin deposits of Pathra-ghat and Mangalhat in Bhagalpur, of Singhbhum, Jubbulpore and Gwalior might have been activated and used, but their utilization for pottery and porcelain goods rules out their use for other purposes.

The bleaching property of clays is due mainly to two factors: colloidal nature, and porosity (capillary action). According to Menard and Mallory<sup>1</sup> the mechanical and electrical properties of the earth are responsible for its bleaching action. Benedict<sup>2</sup> holds that the colloidal matter in the earth first undergoes atmospheric oxidation when its adsorptive properties develop. As to the mechanism of adsorption, there is hardly any unanimity of views. Sloan<sup>3</sup> holds that removal of colouring matter is mechanical while Kobayashi and Yamamoto<sup>4</sup> find that adsorption proceeds quantitatively according to Freundlich's adsorption isotherm formula. Haseman<sup>5</sup> discredits any explanation based on adsorption. According to Jatkar<sup>6</sup>, the clarification by bleaching earth is solely due to the neutralization of electric charge on the colloidal matter. The removal of large quantities of colouring matter cannot be explained by adsorption alone. Puri and coworkers<sup>7</sup> observe that the colour and other suspended impurities are absorbed in the interstices of clay particles and as such creation of increased pore space of optimum size increases bleaching property of clays. The presence of humus fills up the pore spaces between clay particles and reduces adsorbing property. Hence according to Puri and his coworkers<sup>7</sup> one of the criteria for bleaching clay is the presence of the least amount of humus in it. If humus is present, its removal is essential for activation. The dissolution of aluminium and ferric oxides by acid treatment results in the release of more capillary spaces and increased bleaching power. Parekh and Vaidya<sup>8</sup> also support the view that colouring matter is removed by adsorption.

Clays and earths for adsorptive purposes possess certain characteristics. Each and every clay cannot be activated. The

suitability of a clay for activation does not depend on any single characteristic but on a combination of factors, some of which are: (a) high percentage of silica; (b) a rectilinear dehydration curve; (c) an optimum amount of acidity in the clay; (d) base exchange capacity equal to 30 m.e.; (e) negligible quantity of humus; (f) large proportion of conventional clay fraction and very low ultra-clay fraction; (g) specific amount of sesquioxides removable by acid treatment under certain conditions; and (h) montmorillonite type crystal structure under X-rays.

It is, however, unanimously agreed that a soil cannot be activated unless its humus content is negligible and it possesses an optimum quantity of clay fraction and a specific base exchange capacity.

The present work was undertaken to study the suitability of Gangetic silt for activation. As will be evident from the analysis given below the proportion of silica and conventional clay fraction in Gangetic silt is high, and such clays have been known to be amenable to activation.

The process of activation of clays as recommended by previous workers consists of the following stages: (a) acid treatment for part removal of aluminium oxide to obtain a porous structure; (b) washing the acid-treated clay to a definite degree of acidity; and (c) heating to an optimum temperature.

In the present paper the following procedure has been adopted to activate the clay:

(i) Alkali treatment (for removal of humus); (ii) acid treatment (with mineral acid); (iii) washing the acid-treated clay; and (iv) heating the treated clay to an optimum temperature to partially remove water of hydration to give a stable and porous structure to the clay material. (Heating to a very high temperature is not desirable since it may break down the chemical structure.)

### Experimental

Gangetic silt collected at Calcutta after the rainy season has the following composition and characteristics:

	per cent
Free silica	27.7
Clay substance	56.7
CaCO <sub>3</sub>	8.0
Limonite	8.8
Al <sub>2</sub> O <sub>3</sub>	22.4
Fe <sub>2</sub> O <sub>3</sub>	9.8

$\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$  passing into solution on acid treatment (0.5 N HCl for 3 hr.), 16 per cent.

"Humus" passing into solution on treatment with alkali (0.5N NaOH for 3 hr.), after acid treatment, 16.5 per cent.

1 gm. of silt required 65 mg. of NaOH for removal of the humus.

**Dehydration Curve** — The dehydration curve is determined by noting the loss of weight over a range of temperature between 100°-600°C. when a weighed quantity of the clay was heated in an electric furnace (FIG. 1).

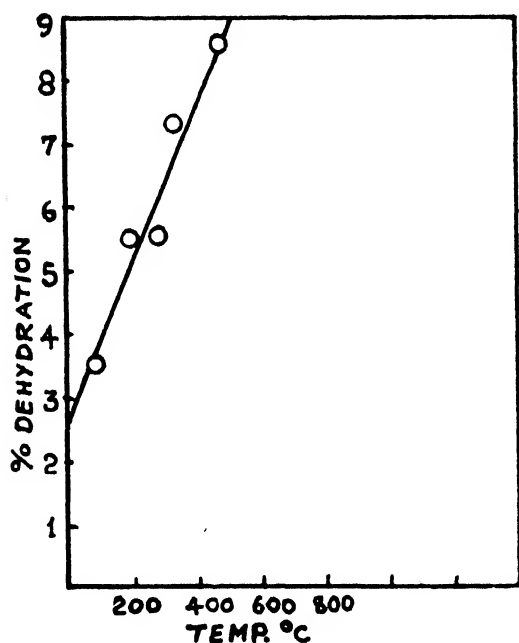


FIG. 1

Gangetic silt contains high silica, low humus and a high clay fraction. It also gives a rectilinear dehydration curve. These are favourable characteristics as will be evident from the following comparison with the analysis of clays activated by previous workers<sup>7</sup>.

TABLE I

CLAY TYPE	$\text{Fe}_2\text{O}_3$ + $\text{Al}_2\text{O}_3$ %	HUMUS %	CLAY SUBS- TANCE %	$\text{Fe}_2\text{O}_3$ + $\text{Al}_2\text{O}_3$ DISSOLVED BY ACID TREATMENT %
Indian bentonite	3.56	0	77.2	3.56
Rawalpindi clay	8.62	0	40.1	...
Puri <sup>7</sup> (P.C. 261)	15.34	0.28	44.3	...
Puri <sup>7</sup> (P.C. 123)	14.78	0	85.5	14.78
Puri <sup>7</sup> (P.C. 72)	15.57	0.44	45.9	15.75
Gangetic silt	32.2	3.5	56.7	16.0

Eight samples of treated Gangetic silt were prepared as follows:

(1) Treated with 3N·NaOH for 4 hr. under reflux, filtered and washed free of alkali.

(2) Treated with 2N·KMnO<sub>4</sub> (alkaline) and washed free of alkali after filtration.

(3) A portion of (1) was treated with HCl, filtered and washed free of acid.

(4) A portion of (2) was treated with HCl, filtered and washed free of acid.

(5) Treated with 4N·HCl under reflux for 4 hr., filtered and washed free of acid.

(6) Treated with 4N·HNO<sub>3</sub> as in (5), filtered and washed free of acid.

(7) Treated with 4N·H<sub>2</sub>SO<sub>4</sub> as in (5), filtered and washed free of acid.

(8) Untreated sample of silt.

The samples prepared as above were divided into two sets. One set of samples was heated at 200°C. for 2 hr. in a tube furnace and the other set of samples was heated at 400°C. for 2 hr. and these were allowed to cool in a desiccator. The samples were stored in well-stoppered bottles for subsequent tests.

The following tests were carried out with the activated samples: (a) determination of specific pore volume; (b) acetic acid adsorption; (c) caramel adsorption; (d) methylene blue adsorption; and (e) oil refining characteristics.

(a) *Specific Pore Volume* — Test pieces were made with each of the samples prepared above and also of the untreated clay. These were slowly heated at 200°C. and 400°C., cooled in a desiccator and weighed in air ( $W_1$ ). The pieces were then left overnight soaked in dry kerosene and again weighed after removing the kerosene film on the surface with a filter paper ( $W_2$ ). Then the pieces were again weighed in kerosene ( $W_3$ ). From these weights the specific pore volume is given by

$$\text{Sp. pore vol.} = \frac{W_2 - W_1}{W_3 - W_2}$$

where

$W_1$  = dry weight,

$W_2$  = weight when soaked in kerosene,

$W_3$  = weight in kerosene of the soaked sample.

The specific pore volumes are given in Table II.

It will be observed that except in the case of permanganate treatment, there is a marked increase in specific pore volume. But this is not the decisive factor, as the size of the pores is also a determining factor



TABLE II

SAMPLE No.	HEATED AT 200°C.,	HEATED AT 400°C.,
	SP. PORE VOL.	SP. PORE VOL.
1	0.49	0.49
2	0.41	0.40
3	0.57	0.55
4	0.53	0.53
5	0.52	0.51
6	0.51	0.54
7	0.51	0.51
8	0.39	0.42

in adsorption and the capillary size is not known here. It is also seen that heating at 400°C. does not appreciably increase the pore space.

(b) *Adsorption of Acetic Acid*—For adsorption of acetic acid, an N/10 solution of acetic acid was employed. About 0.5 gm. of each sample and 25 c.c. of N/10 acetic acid were taken in stoppered bottles and shaken for half an hour in a mechanical shaker and allowed to settle. 10 c.c. of the supernatant were withdrawn and titrated with standard N/10-NaOH solution. The results are given in Table III.

TABLE III

SAMPLE No.	HEATED AT 200°C.,	HEATED AT 400°C.,
	WT. ADSORBED PER GM. OF SAMPLE (GM.)	WT. ADSORBED PER GM. OF SAMPLE (GM.)
1	0.057	0.036
2	0.031	0.016
3	0.018	0.0016
4	0.020	0.0016
5	0.016	0.0014
6	0.019	0.0015
7	0.016	0.0013
8	0.011	0.0041

An examination of the data shows a gradual decrease in the amount of acid adsorbed from sample 1 to 8. It may be concluded that not only the porous structure formed by the removal of humus plays a part in adsorption, but the oxides of iron and aluminium are also partly responsible for adsorption. In case of acid digestion, these oxides get dissolved and hence the decrease in the adsorption capacity of such samples (No. 3 and 7).

Varying the condition of NaOH treatment, the following results were obtained:

TABLE IV

SAMPLE No.	TREATED WITH 2N. NaOH FOR, HR.	TEMP. OF ACTIVATION, °C.	WT. OF ACETIC ACID ADSORBED (GM.)
A	2	200	0.0305
B	2	300	0.0360
C	4	200	0.0385
D	3	200	0.0305
E	3	300	0.0360

It is thus observed that treatment with alkali for greater length of time increases adsorptive capacity.

From Table III it will be seen that if the temperature of heat treatment is increased to 400°C., the amount of adsorption decreases. It is further observed that raising the temperature to 300°C. increases the absorptive quality. There is evidently a structural breakdown beyond 300°C. This is substantiated by the dehydration curve.

(c) *Adsorption of Caramel*—6 gm. of cane sugar were taken in a conical flask and heated in a sulphuric acid bath to 210°C. The caramelized sugar was dissolved in water and made up to 1 litre and rendered slightly acid with dilute hydrochloric acid; 50 c.c. portions of this solution were taken in separate stoppered bottles, each containing differently prepared samples of activated silt. The bottles were put in a shaker for half an hour. A Dubosq colorimeter was used to study the amount of decolourization.

TABLE V

1 gm. of sample + 50 c.c. of caramel solution

SAMPLE No.	HEATED AT 200°C.,	HEATED AT 400°C.,
	DECOLOURIZATION	DECOLOURIZATION
1	nil	nil
2	28.5	nil
3	25.0	26.0
4	10.4	7.7
5	17.8	11.8
6	32.6	5.3
7	30.2	19.0
8	4.1	nil

Table V shows that acid-treated samples are better decolourizing agents.

(d) *Adsorption of Methylene Blue*—1.0875 gm. of methylene blue were dissolved in water and made up to 1 litre. 75 c.c. of this solution were taken, made alkaline with 5 c.c. of N/10-NaOH (methylene blue is better adsorbed in alkaline medium) and made up to 100 c.c.; 50 c.c. portions of this solution were taken in bottles containing different samples. The contents were shaken for half an hour in a mechanical shaker and allowed to settle. The colour of the supernatant was matched with that of the remaining 50 c.c. of methylene blue solution. The results obtained are shown in Table VI.

It will be seen that the acid-treated silt samples are better adsorbents of colour.

TABLE VI

SAMPLE No.	200°C. WITHOUT ALKALI		200°C. WITH ALKALI	
	wt. of sample (gm.)	% decolourization	wt. of sample (gm.)	% decolourization
1	0.53	13.0	0.52	16.6
2	0.54	37.5	...	...
3	0.54	23.0	0.52	66.0
4	0.52	20.0	...	...
5	0.52	23.0	0.52	60.0
6	0.54	23.0	...	...
7	0.53	16.6	...	...
8	0.51	13.0	...	...

The removal by acid of iron and aluminium gives a better porous structure and higher specific pore volume irrespective of the size of pores, and is more responsible for decolourization. It is also observed that electro-positive dyes are more effectively adsorbed than electro-negative dyes.

(e) *Decolourization of Groundnut Oil*...

The decolourization of groundnut oil with mixtures of activated clay No. 3 and "clarit" was studied. 50 c.c. of oil were shaken with each sample for 25 min. at 90°C., cooled and the colour matched with the original oil sample (TABLE VII).

TABLE VII

WT. OF SAMPLE (GM.)	% ADSORPTION OF COLOUR
0.5 clay	25.1
0.4 clay }	26.4
0.1 clarit }	
0.3 clay }	27.5
0.2 clarit }	
0.2 clay }	28.5
0.3 clarit }	
0.1 clay }	28.0
0.4 clarit }	
0.5 clarit	26.8

It will be seen that there is a slight increase in the percentage adsorption in the initial stages which reaches a maximum, and then there is a slight downward trend.

### Discussion

The above results show that the adsorption property displayed by activated clay varies with the nature of the substance to be adsorbed. It is also seen that while acid-

treated silt, which has been previously treated with alkali, is a good adsorbent of colour, the simple alkali-treated clay is a good adsorbent of acetic acid. Here the aluminium oxide and ferric oxide are effectively removed. These perhaps play an important rôle. It is, therefore, difficult to hold that in the case of acetic acid adsorption the removal of alumina and iron oxide is necessary. Humus may choke up pore spaces and so hinder adsorption, but alumina and iron oxide, which form a lyophobic suspension and are easily precipitated by an electrolyte, may not do so. But, for colour adsorption from caramel solution, which takes place better in an acid medium, there may be dissolution of some iron present in the clay and thus impart colour to the solution. Hence, the presence of iron oxide is undesirable. It is not clearly known what part alumina plays in such cases.

From these considerations it may be held that the process of activation must vary with the purpose for which the clay is to be used. Further, though by the removal of humus and sesquioxides the pore space is increased, the size of these pores are not known. For viscous substances a fine capillary is inactive, while for a non-viscid substance the same pore space may be active. Determination of the pore space does not lead to any definite conclusion.

### Acknowledgement

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# A Note on Infra-red Radiation Treatment of Paddy

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**A**BOUT 60 per cent of the rice consumed in India is par-boiled. The par-boiling process as practised in the country is not standardized. The drying of treated paddy, for instance, has not received sufficient attention, and sun-drying, which is widely followed, is time consuming, and does not lend itself to control. There is also the risk of infection by putrefying organisms and the development of objectionable odour in the finished rice. The moisture content of cured paddy also is an important criterion in husking without damage, and has to be controlled.

By shortening the period, and improving the technique of drying cured paddy, the objectionable odour in par-boiled rice can be eliminated and the husking process improved.

The present investigation relates to a comparative study of the water soluble vitamins (B Group), odour, colour and swelling number of samples of par-boiled rice obtained from cured paddy dried by exposure to (1) sun, and (2) infra-red radiation.

## Experimental

A variety of *aman* rice (*Bhasamanik*) was used for this investigation. The equipment used for infra-red exposure consisted of an oven, enclosed on the sides, with top opening for ventilation, and provided with 2 units in series (dull emitters), one above and one below a wire rack on which the material to be dried is spread. The standard rating of each unit is 700 W and the wavelength emitted is 40,000Å. The heating chamber is totally reflective. The oven can be worked either with both lamps on or with one.

3,000 gm. of paddy were steeped in 6 litres of water and then boiled in the steep. The details of treatment are given in Table I.

Portions of the treated paddy were dried by sun-drying and by infra-red radiation.

TABLE I

No.	TEMP. OF STEEP, °C.	DURATION OF STEEP, HR.	TEMP. DURING PAR-BOILING, °C.	DURATION OF BOILING, HR.
1	35	24	80	1.25
2	35	72	80	1.0

TABLE II—RICE FROM PADDY DRIED IMMEDIATELY AFTER PAR-BOILING

	STEEPED FOR 24 HR. FOLLOWED BY 1.25 HR. BOILING		STEEPED FOR 72 HR. FOLLOWED BY 1 HR. BOILING	
	Sun-drying	Infra-red drying	Sun-drying	Infra-red drying
Thiamin content, $\mu\text{gm./gm.}$	1.88	1.85	1.78	1.76
Nicotinic acid content, $\mu\text{gm./gm.}$	31.89	31.80	31.15	30.9
Swelling number	270	267	268	269
Drying time, hr.	12	2.25	12.5	2.35
Final moisture content, per cent	11	10	10	10
Colour	reddish	reddish	cream	cream
Odour	slight	faint	prominent	faint
Average temperature of grains during drying, °C.	45	70	44	77

TABLE III—RICE FROM PADDY LEFT FOR TWO DAYS AFTER PAR-BOILING BEFORE DRYING

	STEEPED FOR 24 HR. FOLLOWED BY 1.25 HR. BOILING		STEEPED FOR 72 HR. FOLLOWED BY 1 HR. BOILING	
	Sun-dried	Infra-red	Sun-dried	Infra-red
Thiamin content, $\mu\text{gm./gm.}$	1.8	1.78	1.76	1.73
Niacin content, $\mu\text{gm./gm.}$	30.4	30.05	29.8	29.4
Swelling number	263	260	261	262
Drying time, hr.	11	2	11.50	2.08
Final moisture content, per cent	11	10	11.25	10.5
Colour	reddish	reddish	reddish	reddish
Odour	prominent	slight	objectionable	prominent

TABLE IV

Thiamin, $\mu\text{gm./gm.}$	0.78
Niacin, $\mu\text{gm./gm.}$	21.7
Swelling number	370
Colour	cream
Odour	nil

The drying time required to bring down the moisture content to the air-dried level (10 per cent) was noted in each case.

The dried samples were husked in a laboratory mill, and thiamin<sup>1</sup>, nicotinic acid<sup>2</sup>, and swelling number<sup>3</sup> determined. The results are shown in Tables II and III.

The characteristics of raw milled rice obtained from the same paddy are given in Table IV.

It will be observed that infra-red radiation reduces the period of drying to a considerable extent without lowering the vitamin value. The space required for drying is also much smaller than in sun drying.

The adoption of radiant drying in par-boiling process may help to eliminate the boiling or wet-heating stage, if the steeping is carried out with water at a higher temperature.

The authors are grateful to Mr. P. B. Sen for his kind encouragement and helpful guidance during the progress of the work.

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## A Simple Volumenometer

E. J. VERGHESE

*Agricultural Research Institute, Coimbatore*

**D**ETERMINATION of the volume of powders like rice flour, powdered coffee, cement, etc., by displacement methods are often vitiated by the solution effects of the displacing liquids. Difficulties of this type have been overcome by the use of volumenometers: Blyth and Blyth<sup>1</sup>, Francis and Oxnard<sup>2</sup>, Binnington *et al.*<sup>3</sup>, Reilly and Rae<sup>4</sup> among others.

With regard to rice, Kondo<sup>5</sup> and Kondo and Okamura<sup>6</sup> determined the volume of raw and cooked rice by water displacement. Srinivasan and Mudaliar<sup>7</sup> improvised an improved dilatometer for the same purpose. Verghese<sup>8</sup>, during the course of an investigation on quality in rice, felt the necessity for measuring the volume of rice, raw and cooked, and of rice flour out of contact with water or other displacing liquids and the apparatus detailed in this paper was consequently devised.

*The Volumenometer* — (A) The volumenometer bottle is a jam jar, 200-300 c.c. capacity, (B) a rubber washer and (C) the inner lid. A short length (6-9 inches) of a narrow metal tube is soldered air-tight over a suitable aperture cut in the centre of (C). The tube then passes through an opening cut on the collar-like outside lid of the jar so that when the jar is closed and the collar tightly screwed on, the bottle is

connected to outside air only through the metal tube (D).

The metal tube (D) is connected by a bent capillary tube (F) to a 100 c.c. pipette bent as shown and fixed to a wooden board by clips. Prior to being fitted upon the board, two marks ( $G_1$ ) and ( $G_2$ ) are made on the straight end portions of the pipette and the volume of the tube in between these two marks established by determining the weight of water contained therein at room temperature. The lower end of the pipette is connected by rubber tubing to a y piece (H). The other two ends of the y piece are connected to a manometer. This is formed by a glass tube (I) fixed on the board and a rubber tube (J) attached to a reservoir of mercury (K). Readings of the manometer are made on the scale (L) screwed on to the board. Two meter scales are placed end to end so that their zero points coincide. On one side of the scale are the straight end portions of the pipette tube and on the other the glass tube arm of the manometer.

(M) is a small bottle containing a known volume of distilled water, a provision for creating aqueous tension of saturated water vapour when the apparatus is in use.

*Volume of the Volumenometer Bottle* — This is determined as follows:

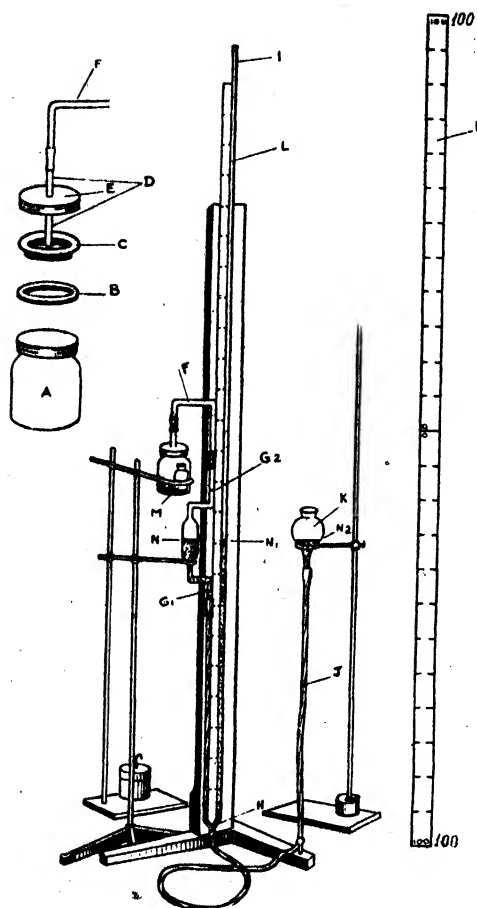


FIG. 1 — A SIMPLE VOLUMENOMETER.

*Step I*—To start with, the mercury reservoir is raised or lowered until the pipette tube is about half filled with mercury—approximately at position N-N<sub>1</sub>-N<sub>2</sub>. The reservoir is clamped in this position for a while. When the mercury level at N<sub>1</sub> is constant, the volumenometer bottle is attached and closed air-tight. A certain mass of air is thus enclosed at room temperature and atmospheric pressure.

*Step II*—The reservoir is then lowered until the mercury level stands at the mark G<sub>1</sub> of the pipette. The enclosed air thus expands in volume and consequently there is a fall in pressure. The difference in pressure is noted. The reservoir is again raised to its initial position and once more brought down and a second reading of pressure made. The average of the two readings is taken for calculation. Let the difference in pressure be P<sub>1</sub>. Then, the room temperature remaining constant, according to Boyle's Law,

$$(V+V_1)(B-P_1-W)=K \quad (1)$$

Where

V = volume of the volumenometer bottle, i.e. volume of air contained in the bottle plus the connecting tube up to the mark G<sub>2</sub> of the pipette

V<sub>1</sub> = volume of the pipette tube = volume from mark G<sub>2</sub> to G<sub>1</sub>

B = barometric pressure at room temperature

P = difference in pressure due to the rarefaction

W = tension of saturated water vapour at room temperature

*Step III*—The reservoir is then raised to the upper mark G<sub>2</sub>. The enclosed air is compressed and hence the mercury level in the manometer tube rises. The increase in pressure is noted. The reservoir is again brought down, then raised to the mark G<sub>2</sub> and another reading for increase in pressure taken. The average of the two readings is noted. Let this be P<sub>2</sub>. Then

$$V(B+P_2-W)=K \quad (2)$$

From equations (1) and (2), we get

$$(V+V_1)(B-P_1-W) = V(B+P_2-W) \quad (3)$$

$$V = \frac{V_1(B-P_1-W)}{P_1+P_2}$$

V<sub>1</sub>, P<sub>1</sub>, P<sub>2</sub> are already known. B is read on a standard barometer fitted up in the same room and W is read from tables. V can then be calculated. A number of separate determinations are made and the average value taken as the volume of the bottle. The results of two sets of determinations are given in Tables I and II.

TABLE I — VOLUME OF VOLUMENOMETER BOTTLE NO. 1

V <sub>1</sub>	B	W	P <sub>1</sub>	P <sub>2</sub>	V = $\frac{V_1(B-P_1-W)}{P_1+P_2}$ c.c.
100	72.510	3.9565	9.40	8.5	330.5
100	72.475	3.9565	7.10	11.5	330.2
100	72.400	4.0007	11.40	11.4	331.4
100	72.410	3.9560	7.50	10.9	331.3
100	72.500	3.9340	4.90	14.4	329.9
100	72.450	3.8045	3.35	16.35	331.4
100	72.500	3.8045	7.45	11.05	331.1
AVERAGE					330.8

TABLE II — VOLUME OF VOLUMENOMETER BOTTLE NO. 2

		W		P <sub>1</sub>		V <sub>1</sub> (B - P <sub>1</sub> - W) c.c.
						P <sub>1</sub> + P <sub>2</sub> c.c.
100	72.595	3.5760	12.45	15.10		205.3
100	72.730	3.7410	12.46	15.04		205.5
100	72.550	3.6165	12.25	15.35		205.3
100	72.085	2.9441	12.98	14.30		205.9
100	72.335	3.4761	13.28	15.55		205.7
100	72.630	3.0479	12.75	15.02		204.9
100	72.350	3.0480	12.85	14.58		205.8
100	72.380	3.1190	12.90	14.50		205.6
100	72.460	3.0131	13.00	14.45		205.6
100	72.480	2.9782	13.30	13.98		206.0
100	73.085	3.0479	11.48	17.00		205.6
100	72.890	2.7290	11.25	17.47		205.2
AVERAGE						205.5

TABLE III

No.	WEIGHT GM.	VOLUME	
		Raw, c.c.	Cooked*, c.c.
1	20	13.5	31.1
2	20	15.0	31.1
3	20	14.9	32.8
4	20	15.4	31.6
5	20	14.5	40.4
6	20	14.9	30.5
7	20	14.3	30.8
8	20	14.0	30.2
9	20	14.7	31.1
10	20	14.9	31.1
11	20	14.6	33.2
12	20	14.8	35.6
13	20	15.0	34.3
14	20	14.0	33.6
15	20	14.1	33.8
16	20	14.6	35.7
17	20	14.6	36.8
18	20	14.8	37.3
19	20	14.0	37.1
20	20	14.8	38.2
21	20	13.9	37.9
22	20	13.7	38.0
23	20	14.3	39.3
24	20	14.1	39.1
25	20	14.1	40.2
26	20	14.3	44.4
27	20	14.3	41.2
28	20	14.3	54.5
29	20	14.2	53.6
30	20	14.0	54.6
31	20	14.7	51.8
32	20	14.5	57.7
33	20	14.6	67.7
34	20	14.6	63.1
35	20	14.9	60.9
36	20	14.0	59.1
37	20	14.4	53.2
38	20	13.8	67.4
39	20	13.5	66.1
40	20	14.1	69.7
41	20	14.7	66.0
42	20	13.9	64.6

\* After reading the volume in the volumometer, the rice is cooked for different intervals of time and the volume of cooked rice determined. The results are given in column 4.

**Volume of Sample**—The sample of rice or any other material, the volume of which is to be determined, is carefully introduced into the volumometer bottle at step I before it is clamped and closed air-tight. Steps II and III are repeated just as before. In other words the volume of the volumometer bottle is once more determined. The difference in the two determinations will give the volume of the sample.

The accuracy of the apparatus was tested by estimating the volume of known weights of glass beads, lead shots, etc. Results obtained were concordant.

During the last six years, several hundreds of samples of rice were examined for the gain in volume on cooking by the method detailed above. Results of the volume measurements of forty-two 20 gm. aliquot portions of a uniformly grained sample of hand-pounded rice GEB 24 are given in column (3) of Table III. A statistical analysis of the data gave a low standard deviation, 0.41, and a low coefficient of variation 2.84, testifying to the reliability and accuracy of the method.

#### Acknowledgement

The author is deeply indebted to Mr. P. D. Karunakar, Government Agricultural Chemist, Madras, for affording facilities for conducting the work and for helpful suggestions and criticism.

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## Letters to the Editor

### CHEMICAL EXAMINATION OF THE LATEX FROM EUPHORBIA TIRUCALLI

KARIMULLAH AND DUTTA<sup>1</sup> REPORTED THE isolation of a crystalline ketone from the dried latex of *Euphorbia tirucalli*. Karim-

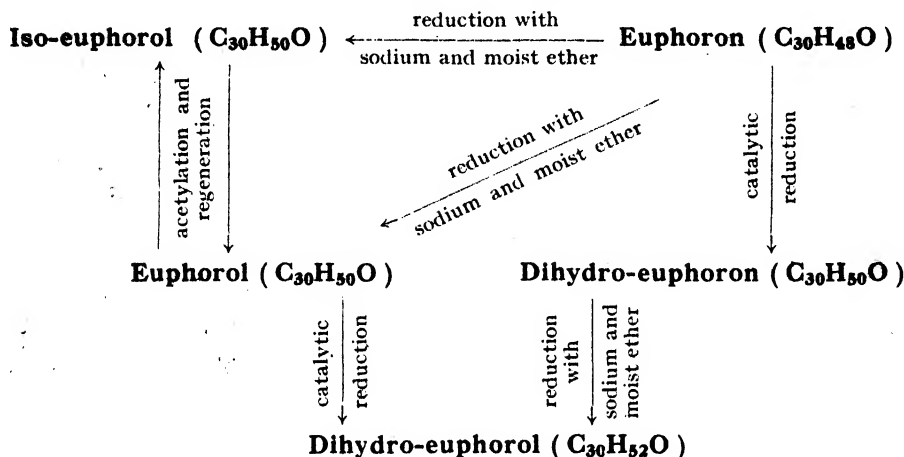
ullah and Gopalachari<sup>2</sup> subsequently named this ketone euphoron,  $C_{30}H_{48}O$ , and showed that it forms the main crystalline constituent of the dried latex.

Working on a sample of the fresh latex of *Euphorbia tirucalli* recently received from South India in a sealed tin, a new

crystalline substance has been isolated in a yield of 5 per cent (on the weight of dry matter) in place of the ketone isolated earlier (*loc. cit.*). This substance is an alcohol, m.p., 118°-119°C.,  $[\alpha]_D^{20} = +36.5$  which analyses for the formula  $C_{30}H_{50}O$  (found: C, 84.0; H, 11.94; calculated for  $C_{30}H_{50}O$ ; C, 84.41; H, 11.74) and has been provisionally

of the two borneols from camphor by reduction with sodium and alcohol.

Results of further studies in the constitution of euphoron and the various products derived from it are being communicated in a separate paper<sup>3</sup>. The relationship between these products and euphoron may be represented as follows:



named *iso*-euphorol. It is isomeric with euphorol,  $C_{30}H_{50}O$ , m.p., 108°-109°C.,  $[\alpha]_D^{20} = +24$ , obtained from euphoron by reduction with sodium and moist ether<sup>3</sup>. Its acetyl derivative as well as the product of its catalytic reduction are identical with euphorol acetate,  $C_{32}H_{52}O_2$ , m.p., 107°-108°C., and dihydro euphorol,  $C_{30}H_{52}O$ , m.p., 122°-123°C. respectively. Euphorol acetate, on hydrolysis with N/10 alcoholic potash, yields a crystalline product from which both euphorol and *iso*-euphorol have been obtained by fractional crystallization. Further, it has been noted that when euphoron dissolved in the minimum quantity of moist ether is reduced with a large excess of sodium, the resultant product gives, after a series of fractional crystallizations, euphorol as the major fraction of the total crystallizate, and a small quantity (2 per cent) of a substance which melts at 118°-119°C., shows no depression in m.p. when mixed with *iso*-euphorol and gives euphorol acetate on treatment with acetic anhydride. It would thus appear that *iso*-euphorol is also produced along with euphorol in the reduction of euphoron with sodium and moist ether and that these two alcohols are stereo-isomerides derived from the same ketone euphoron. The formation of these isomeric alcohols in the reduction of euphoron is somewhat similar to the formation

#### Acknowledgement

The authors thank the District Forest Officer, Madura for supplying the latex used in these investigations.

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*Chemical Laboratories, Council of Scientific & Industrial Research, Delhi*  
May 9, 1949

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#### OCCURRENCE & UTILIZATION OF VANADIUM-BEARING IRON ORE IN BIHAR

OVER A STRETCH OF 30 MILES FROM BARIARPUR railway station (Sahibganj Loop, E.I.R.) running more or less along the Monghyr-Jamooee Road, up to the Jamooee railway station (E.I.R. Main Line) and close to the eastern slopes of the hills lie partially smelted vanadium-bearing iron ore deposits. They are concentrated at Mainma Tal near the Abhaipur railway station. Large dumps are seen at Khotadih and Bhaduridih and Kharagpur Lake in the low-lying fields. At Ghorakhpur, Pankul, Lakhon Sukhmaria, Kusmaha, Jogti, Kadarkol and Kauba, they

occur in hilly regions. The first is easily accessible from Shampur, and the others from Bharhat, 5 miles east of the Jamooee railway station. The ore bodies appear to be associated with schists at the out-crop near the village Nauagarhi, 5 miles west of Monghyr, and lie in dumps near the mines worked long ago. These can be worked side by side with the dumps. However, the dumps are large enough to give all the mineral required for several years to come.

Partially smelted ore bodies have been also observed about 70 miles from this zone at Ranijharna close to the village Lohsingha in the Banka sub-division of Bhagalpur district. The present investigations are confined to Benaili Zamindari only, but similar ores may occur in the areas of other zamindaris, particularly, the Lachmipore Zamindari.

Analysis of a representative sample of the ore from Benaili Zamindari gave the following results:

SiO <sub>2</sub>	26.60	per cent
Al <sub>2</sub> O <sub>3</sub>	12.67	
Fe <sub>2</sub> O <sub>3</sub>	56.67	
TiO <sub>2</sub>	0.71	
MnO <sub>2</sub>	1.95	
V <sub>2</sub> O <sub>3</sub>	0.1-0.5	
Undetermined	1.45-0.95	

On the basis of this analysis, the following conclusions appear justified: (1) the vanadium content of the ore is not high enough to hold out prospects for the extraction of vanadium oxide; (2) direct manufacture of suitable alloys from the ore by suitable treatment is a feasible alternative. The titanium content of the ore is low and is unlikely to interfere with the smooth working of the blast furnace; it can be removed easily along with the slag. The vanadium content is high enough to produce the desired alloys (vanadium steels contain V<sub>2</sub>O<sub>3</sub> varying from 0.11 to 0.18); (3) the silica (26.6 per cent) can be removed completely during the smelting operation. The elimination of silica raises the vanadium content by 26.6 per cent.

A careful survey of the whole area has to be made and further samples have to be analysed in order to establish the suitability of the ore for the direct manufacture of vanadium steels. Jamalpur is a convenient site for erecting the smelting furnaces. The nearest dump is 6 miles away from it and cheap power is available for running electric furnaces.

*Bhagalpur*  
April 30, 1949

A. N. CHOWLA

## IONOSPHERIC ABNORMALITIES DURING SOLAR DISTURBANCES

IN COURSE OF OUR ROUTINE OBSERVATIONS on ionosphere carried out at Calcutta, certain abnormal characteristics of the E and the F regions have been observed since the middle of January 1949. These appear to be related to the pronounced increase in sun-spot activity noticed since the same time. The purpose of the present note is to record the salient features of these ionospheric abnormalities.

The days in which the abnormal disturbances occurred were more frequent in February than in January. The disturbed days in January were: 17, 18, 20, 21, 26 and 27. In February almost all the days throughout the month excepting those in the first week were found to be disturbed. On January 26 and on February 16, the disturbances were very pronounced and were accompanied by radio fade-outs. The other days were only moderately disturbed.

During the days of moderate disturbance, the ionospheric characteristics showed abnormal variations, but reflections could be obtained throughout the hours of observations. The general nature of variations during such days was as follows: the ionization of region E increased abnormally, specially in the afternoon and in the early part of the night. The ionization of region F<sub>2</sub> during daytime did not deviate appreciably from the normal trend, but the values of  $y_m$  (vertical semi-thickness of the layer assuming parabolic distribution of ionization) and  $h_{max}$  (height of maximum ionization) were found to be higher than normal. At night, however, the ionization of this region attained abnormally high values while the values of  $y_m$  and  $h_{max}$  came down nearly to normal.

During days of pronounced disturbance, January 26 and February 16, the abnormalities were very great, so much so that during certain periods reflections from all ionospheric layers ceased altogether. In other words, fade-outs occurred. The nature of variations on these days for the different ionospheric regions is discussed below.

On January 26, the ionization of region E suddenly began to increase from 10.45 hr. and very high frequencies were reflected from this region. Reflections from region F<sub>2</sub> could not be observed from 11.00 hr. On the low frequency side, reflections could not be obtained on frequencies on which



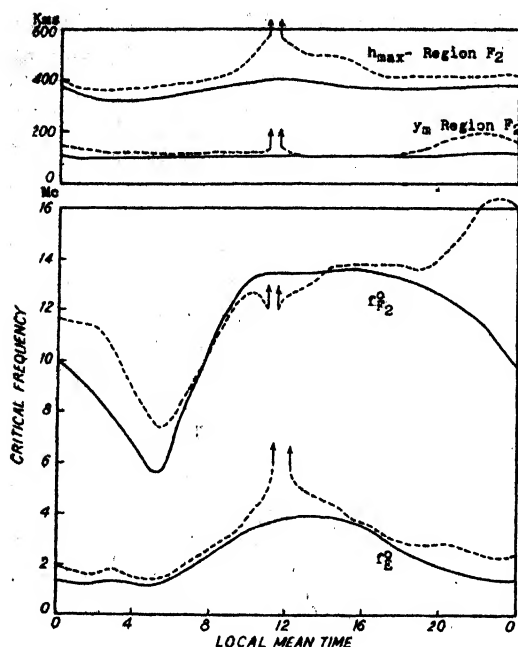


FIG. 1 — VARIATION OF THE CRITICAL PENETRATION FREQUENCY OF REGIONS E AND F<sub>2</sub> (BOTTOM CURVE) AND OF THE THICKNESS AND HEIGHT OF MAXIMUM IONIZATION OF REGION F<sub>2</sub> (TOP CURVE) ON NORMAL DAYS IN JANUARY 1949 (CONTINUOUS LINE CURVE) AND ON THE DAY OF FADE-OUT (BROKEN LINE CURVE). ON THE DAY OF FADE-OUT (26TH JANUARY), REFLECTIONS FROM REGION F<sub>2</sub> STOPPED AT 11.00 HR. AND REAPPEARED AT 11.30 HR. REFLECTIONS FROM REGION E STOPPED AT 11.12 HR. AND REAPPEARED AT 12.12 HR.

E region reflections are normally obtained. Evidently this was due to the formation of a strongly absorbing lower (D) region. About 12 minutes later all reflections ceased. This complete fade-out lasted for about 24 minutes, and reflections began to reappear first from region F<sub>2</sub> at 11.30 hr. and then from region E at 12.12 hr. (FIG. 1).

On February 16, the ionization of region E suddenly began to increase abnormally from about mid-day, and at 13.06 hr. reflections from region F<sub>2</sub> could not be obtained. At 13.30 hr. reflections from both the regions ceased altogether. This fade-out lasted for one hour and a half. Echoes were again obtained first from region F<sub>2</sub> at 15.00 hr. and then from region E at 17.00 hr. (FIG. 2).

On both the days, just before the fade-out of F<sub>2</sub> region reflections, the ionization of this region began to decrease while y<sub>m</sub> and h<sub>max</sub> both increased enormously till reflections from this region ceased altogether

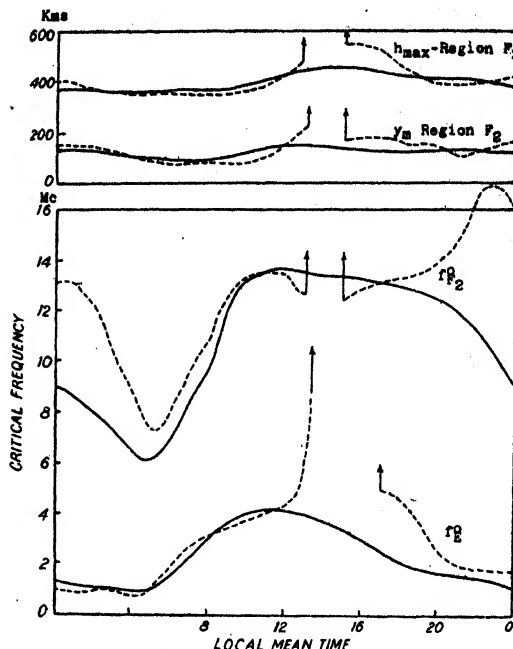


FIG. 2 — THE CURVES DEPICT SIMILAR VARIATION AS FOR FIG. 1. CONTINUOUS CURVE FOR NORMAL DAYS, FEBRUARY 1949. BROKEN LINE CURVES FOR FEBRUARY 16, 1949, ON WHICH FADE-OUT OCCURRED.

(FIGS. 1 and 2). The frequencies which were normally returned from this region were then found to be reflected from E region heights.

About 7 to 8 hr. after the fade-out, the ionization of region F<sub>2</sub> began to increase steadily and on both the days attained a maximum value round about midnight. This value was found to be almost 200 per cent of the normal value. About 2 to 3 hr. after the fade-out, the intensity of the reflections began to increase steadily suggesting the gradual disappearance of the strongly absorbing D region. During the nights following the disturbed days, region E ionization was found to be abnormal (FIGS. 1 and 2).

Investigations described above form part of the programme of the Radio Research Committee of the Council of Scientific & Industrial Research, Government of India, and have been carried out in the Ionosphere Laboratory of Prof. S. K. Mitra.

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R. K. MITRA

*Ionosphere Laboratory  
University College of Science, Calcutta  
May 13, 1949*

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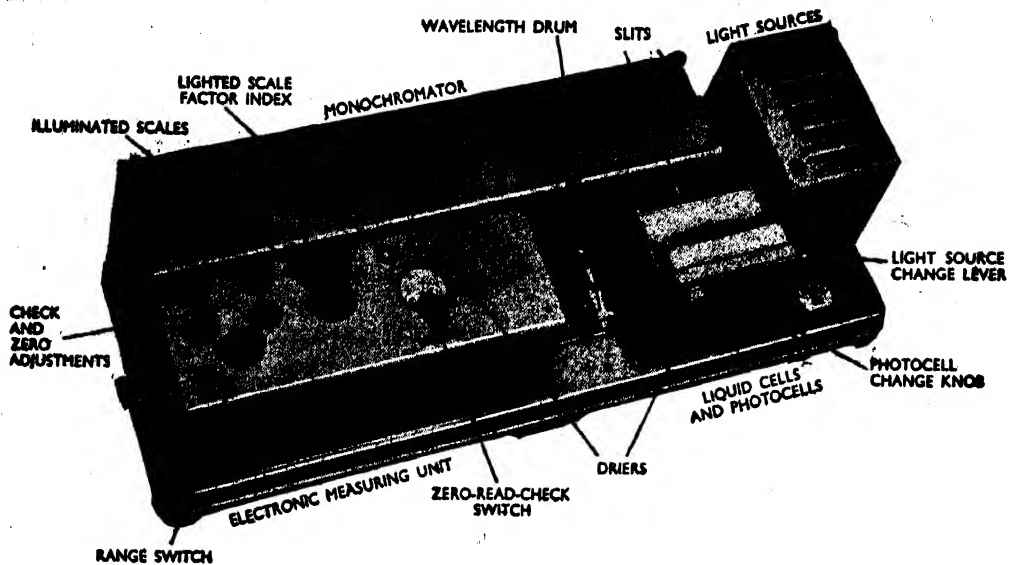
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# The Analytical Uses of Dithiocarbamido-hydrazine

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A NUMBER of sulphur-containing organic compounds are now known to give deeply coloured complexes with bismuth. Mention may be made of thiourea<sup>1</sup>, dimercaptothiodiazole<sup>2</sup>, diethyl dithio-carbamate<sup>3</sup>, and dithizone<sup>4</sup>, based on which methods have been developed by different workers to estimate bismuth colorimetrically when the coloured complexes are soluble either in water or in solvents like chloroform or carbon tetrachloride, or by peptization in aqueous solutions if the complex is insoluble.

The colour which solutions containing bismuth develop with thiourea, a reagent which commends itself as one of the simplest and easily available among organic sulphur compounds, is largely dependent on the concentration of the reagent and other factors, and was for a long time regarded as unsuitable for colorimetry, until Mahr<sup>1</sup> found the limited range of working conditions in which the reaction could be useful.

The present work relates to some analytical applications of the compound dithiocarbamido-hydrazine, a simple derivative of thiourea on which no work appears to have been done, though some complex derivatives of thiourea have been examined. Some characteristic reactions of the compound with metallic cations are now noted and the possibility of utilizing them for quantitative estimation of metals and of their separation from analogous cations from which separation by the classical methods is known to be difficult, is examined.

A summary of the characteristic reactions observed with some of the commoner metals is given in Table I.

TABLE I

METALLIC ION IN SOLUTION	REACTIONS WITH THE REAGENT		
	In acid solution	In neutral solution	In ammoniacal solution
Ag <sup>+</sup>	White heavy bulky ppt., darkens on strong heating. Insol. in acetic acid. Quantitative	White ppt.	Brown, heavy, bulky ppt. Quantitative
Bi <sup>+++</sup>	Deep yellow colouration. No ppt.	...	...
Cd <sup>++</sup>	No ppt.	No ppt.	Fine-grained, white ppt., dissolves in acetic acid. Quantitative
Co <sup>++</sup>	No ppt.	No ppt.	Brown, voluminous, heavy, ppt., dissolves in acetic acid. Quantitative
Cu <sup>++</sup>	White, granular, heavy ppt.	Black, fine-grained ppt., insol. in acetic acid. Quantitative	Black ppt.
Hg <sub>2</sub> <sup>++</sup>	Black, granular, heavy ppt., insol. in acetic acid. Quantitative	Black ppt.	...
Hg <sup>++</sup>	White, granular, heavy ppt., insol. in acetic acid. Quantitative	White ppt.	...
Ni <sup>++</sup>	No ppt.	No ppt.	Chocolate brown, voluminous, heavy ppt., dissolves in acetic acid. Quantitative
Pb <sup>++</sup>	No ppt.	Yellow, granular, heavy ppt., dissolves in acetic acid. Quantitative	
Zn <sup>++</sup>	No ppt.	No ppt.	White, granular ppt., dissolves in acetic acid. Quantitative

The similarity of the reagent with thiourea and the thiocyanates is evident from the observations recorded in Table I. The black compound of copper obtained from acetic acid solution is strongly paramagnetic and is presumably the cupric derivative. The white compound obtained from strongly acidic solutions is, on the other hand, diamagnetic, and is thus a cuprous compound. Both precipitations are quantitative, but the white granular cuprous compound settles and filters easily and is thus more useful from the analytical point of view. Estimation by direct weighing of the precipitates was not possible, because neither of them bears a constant composition. They were decomposed by nitric acid and the copper titrated iodometrically. The cadmium complex, soluble in acids but insoluble in ammonia, also settles and filters easily, showing no signs of oxidation, but had to be decomposed and weighed as  $\text{CdSO}_4$ . A complete separation of copper from cadmium was effected by a single precipitation of the cuprous derivative from strongly acid solutions, and precipitating the cadmium complex from the filtrate by rendering it ammoniacal in presence of a sufficient quantity of the reagent.

Bismuth gives an yellow colour with the reagent stable below an acid concentration of 0.03N, turning turbid on long keeping. The depths of colour with varying concentrations of bismuth were examined and found to be proportional, and independent of the amount of reagent present in excess. The range from 40 mg. to 0.4 mg. Bi per 25 c.c. has been followed by three sets of standards.

### Experimental

The reagent was prepared by the method of Freund and Wischewinsky<sup>6</sup> in the form of white needle-shaped crystals from hydrazine sulphate (10 gm.) and ammonium sulphocyanide (15 gm.). The crystals were recrystallized from hot water (m.p.,  $208^\circ\text{--}210^\circ\text{C}$ ., yield 5-6 gm.). Sulphur was determined by peroxide fusion (found: S, 43.23 per cent;  $\text{C}_2\text{H}_4\text{N}_4\text{S}_2$  requires: S, 42.66 per cent).

*Cupric Dithiocarbamido-hydrazine* — This was prepared in the form of a fine, black powder from a solution of copper sulphate (1 mol.) and dithiocarbamido-hydrazine (1 mol.), the latter being added to a solution of copper sulphate containing 2 c.c. of glacial acetic acid per 100 c.c. of solution. Precipitation

was carried out from warm solution with constant stirring using a slight excess of a 2 per cent solution of the reagent. The product was thoroughly washed with warm water containing a few drops of dilute acetic acid; dried in air and finally in a vacuum desiccator. Copper was estimated iodometrically after decomposition by concentrated nitric acid. Sulphur was estimated by peroxide fusion method (found: Cu, 27.30; S, 28.55; Cu: S :: 1.00: 2.07;  $\text{C}_2\text{H}_4\text{N}_4\text{S}_2\cdot\text{Cu}\cdot\text{H}_2\text{O}$  requires: Cu, 27.70; S, 28.00 per cent).

The substance was paramagnetic. The gram-molecular susceptibility, found with a magnetic balance of the Guoy type, was found to be 1.41 Bohr's magneton, without diamagnetic correction for the constituents ( $l=8.8$  cm.,  $w=0.2708$ g,  $\Delta w=5.97$  mg.,  $H_{\text{max}}=10.4\times 10^3$  gauss).

*Cuprous Dithiocarbamido-hydrazine* — This was obtained as a white precipitate by adding a 2 per cent solution of the reagent to a warm solution of copper sulphate in 2N hydrochloric acid containing 1 gm. of sodium sulphite for every gram of copper. The precipitate was filtered through a Buchner funnel and washed with warm water containing a few drops of dilute hydrochloric acid. The free acid was finally removed by washing with absolute alcohol.

The product dried in a vacuum desiccator contains an appreciable quantity of free sulphur which could be isolated by extraction with carbon disulphide.

The substance was examined in a Curie balance and found to be diamagnetic.

*Cadmium Dithiocarbamido-hydrazine* — Cadmium is precipitated quantitatively as a white fine-grained powder from ammoniacal solutions by the reagent. The reagent was first added to an acidic solution of the cadmium salt which was subsequently made ammoniacal by dropwise addition of strong ammonia with constant stirring. The product after washing with water and drying in a vacuum desiccator to constant weight was analysed. Cadmium was estimated as sulphate by decomposition with concentrated nitric and sulphuric acids, and sulphur was estimated by peroxide fusion (found: Cd, 40.85 per cent, S, 25.4 per cent; Cd: S :: 1.0: 2.17). The compound was found unsuitable for estimation of cadmium by direct weighing.

*Estimation of Copper* — The black cupric compound was precipitated from a warm solution acidified with 2 c.c. glacial acetic

acid per 100 c.c. of the liquid with a freshly prepared 2 per cent solution of the reagent. The precipitate, after settling for sometime on the water-bath was filtered and washed with warm water containing a few drops of dilute acetic acid. It was then decomposed with concentrated nitric acid and estimated iodometrically. The results are given in Table II.

TABLE II

COPPER TAKEN GM.	COPPER FOUND GM.	ERROR
0.1401	0.1403	+0.0002
0.0933	0.0930	-0.0003
0.0200	0.0202	+0.0002
0.0700	0.0696	-0.0004

The white cuprous compound is a heavy granular precipitate and more suitable for analytical work. It was obtained by precipitating a hot solution of copper containing one-fifth of its volume of concentrated hydrochloric acid and 1 gm. of sodium sulphite. The liquid filters rapidly and the precipitate, after thorough washing with hot acidulated water, was decomposed with concentrated nitric acid and the copper estimated iodometrically. On long standing, the precipitate has a tendency to turn grey. The results are given in Table III.

TABLE III

COPPER TAKEN GM.	COPPER FOUND GM.	ERROR
0.1120	0.1116	-0.0004
0.0467	0.0466	-0.0001
0.1401	0.1402	+0.0001
0.2102	0.2096	-0.0006
0.0560	0.0562	+0.0002

*Separation of Copper from Iron* — Copper was precipitated from 2N hydrochloric acid solution as previously described. The filtrate was oxidized with bromine water and iron precipitated as hydroxide with ammonia and ignited to oxide. The results are given in Table IV.

TABLE IV

COPPER TAKEN GM.	IRON TAKEN GM.	COPPER FOUND GM.	ERROR	IRON FOUND	ERROR
0.1455	0.0930	0.1452	-0.0004	0.0931	+0.0001
0.2182	0.0270	0.2176	-0.0006	0.0280	+0.0001
0.2010	0.0557	0.2005	-0.0005	0.0554	-0.0003
0.1455	0.1440	0.1457	+0.0002	0.1436	-0.0004
0.0873	0.3901	0.0875	+0.0002	0.3900	-0.0001
0.0582	0.5574	0.0580	-0.0002	...	...

*Separation of Copper from Zinc* — Copper was separated as the cuprous complex as

before, and the zinc was estimated in the filtrate after treatment with bromine water as zinc ammonium phosphate. The results are given in Table V.

TABLE V

COPPER TAKEN GM.	ZINC TAKEN GM.	COPPER FOUND GM.	ERROR	ZINC FOUND GM.	ERROR
0.0291	0.3120	0.0289	-0.0002	0.3132	+0.0003
0.2843	0.0447	0.2840	-0.0003	0.0447	nil
0.2843	0.0223	0.2846	+0.0003	0.0222	-0.0001
0.0582	0.2682	0.0584	+0.0002	0.2673	-0.0004
0.2910	0.0536	0.2909	-0.0001	0.0534	-0.0002
0.1445	0.1341	0.1452	-0.0003	0.1338	-0.0003
0.0582	0.5360	0.0581	-0.0001	...	...

*Estimation of Cadmium* — A slight excess of a 2 per cent solution of the reagent was added to an acidified solution of the cadmium salt followed by dropwise addition of 5N ammonia with constant stirring. The heavy white precipitate was washed with hot water containing a few drops of ammonia. It was dried in the oven and decomposed to cadmium sulphate with concentrated nitric and sulphuric acids, and weighed as  $\text{CdSO}_4$ . The results are given in Table VI.

TABLE VI

CADMIUM TAKEN GM.	$\text{CdSO}_4$ OBTD. GM.	CADMIUM FOUND GM.	ERROR
0.0804	0.1483	0.0800	-0.0004
0.0832	0.1540	0.0830	-0.0002
0.1110	0.2065	0.1114	+0.0004
0.0555	0.1024	0.0552	-0.0003
0.1665	0.3090	0.1666	+0.0001

*Separation of Copper from Cadmium* — The cuprous compound was precipitated from 2N hydrochloric acid solution and estimated iodometrically as before. The filtrate, after further addition of the reagent, was rendered ammoniacal to precipitate the cadmium compound which was finally decomposed and weighed as  $\text{CdSO}_4$ . The results are given in Table VII.

TABLE VII

COPPER TAKEN GM.	CADMIUM TAKEN GM.	COPPER FOUND GM.	ERROR	CADMIUM FOUND GM.	ERROR
0.0970	0.1110	0.0968	-0.0002	0.1107	-0.0003
0.0200	0.1110	0.0201	+0.0001	0.1106	-0.0004
0.0291	0.1665	0.0292	+0.0001	0.1666	+0.0001
0.2910	0.0277	0.2909	-0.0001	0.0275	-0.0002
0.1460	0.0277	0.1455	-0.0005	0.0274	-0.0003
0.2910	0.0555	0.2913	+0.0003	0.0551	-0.0004

*Colorimetric Estimation of Bismuth* — Pure bismuth solutions only have been examined in the present investigation. Three standard solutions were prepared to cover the whole

range of working concentrations from purest basic bismuth nitrate freshly recrystallized from hot dilute nitric acid. Known volumes of standard solutions were evaporated in a crucible which was ignited at a low temperature and bismuth content determined as oxide. Freshly prepared aqueous solutions of the recrystallized reagent (0.5 per cent) were used, and the colour examined without delay in a Helige Duboscq colorimeter. In one set of experiments the colour was measured in a Lumetron photoelectric colorimeter

TABLE XI — LUMETRON MEASUREMENTS

Concentration of standard bismuth solution, 0.574 mg./c.c.; reagent used, 10 c.c. of 0.5% solution; total volume (83 c.c.) same in all cases.

C.C. BISMUTH SOLUTION	% TRANSMISSION (LUMETRON READINGS)	O.D. $\times 10^4$
1.0	100	0
1.5	87	6
2.0	74	13
2.5	64	19
3.0	54.5	26
3.5	46.5	33
4.0	41	39
4.5	36	44

TABLE VIII

Concentration range of bismuth, 40.35–6.725 mg./25 c.c.; concentration of reagent used, 0.5%; concentration of standard, 16.81 mg./25 c.c.; fixed reading of "unknowns", 20.0 mm.

READINGS OF STANDARD (MEAN OF 3)	CONCENTRATION OF "UNKNOWN" BY BEER'S LAW IN MG./25 C.C.	ACTUAL CONCENTRATION IN MG./25 C.C.
50.50	42.45	40.35
41.74	35.10	33.62
37.50	31.52	30.26
33.08	27.81	26.90
28.72	24.15	23.54
24.40	20.51	20.17
20.06	16.86	16.81
15.68	13.18	13.45
13.49	11.84	11.76
11.25	9.46	10.08
9.10	7.65	8.40
6.89	5.83	6.72

TABLE IX

Concentration range of bismuth, 6.725–1.345 mg./25 c.c.; concentration of reagent used, 0.1%; concentration of standard, 3.862 mg./25 c.c.; fixed reading of "unknowns", 20.0 mm.

READINGS OF STANDARD (MEAN OF 3)	CONCENTRATION OF "UNKNOWN" BY BEER'S LAW IN MG./25 C.C.	ACTUAL CONCENTRATION IN MG./25 C.C.
41.78	7.02	6.72
37.48	6.28	6.05
33.00	5.54	5.38
28.68	4.82	4.70
24.40	4.10	4.03
20.11	3.37	3.36
15.70	2.63	2.69
13.59	2.28	2.35
9.00	1.51	1.68
7.01	1.17	1.34

TABLE X

Concentration range of bismuth, 1.845–0.4035 mg./25 c.c.; concentration of reagent used, 0.05%; concentration of standard, 0.6725 mg./25 c.c.; fixed reading of "unknowns", 20.0 mm.

READINGS OF STANDARD (MEAN OF 3)	CONCENTRATION OF "UNKNOWN" BY BEER'S LAW IN MG./25 C.C.	ACTUAL CONCENTRATION IN MG./25 C.C.
41.70	1.40	1.34
37.29	1.25	1.31
33.01	1.11	1.07
28.70	0.96	0.94
24.41	0.82	0.80
20.00	0.67	0.67
17.80	0.60	0.60
15.69	0.52	0.53
11.39	0.38	0.40

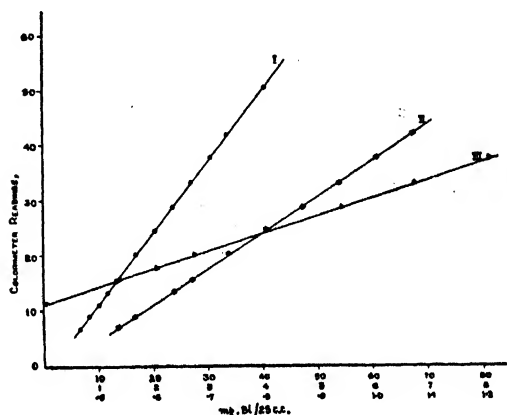


FIG. 1

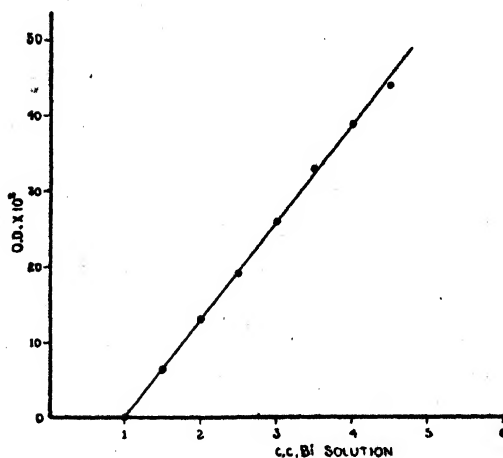


FIG. 2

EF using the blue filter. The results are given in Tables VIII, IX, X and XI.

The results of Tables VIII, IX and X are plotted graphically in Fig. 1, and of Table XI in Fig. 2. The variation, it will be seen, is linear.

## Summary

Dithiocarbamido-hydrazine,  $\text{NH}_2\text{CSNH.NHCSNH}_2$ , a simple derivative of thiourea, has been found to precipitate copper and cadmium quantitatively in acid and ammoniacal solutions respectively, and to give an orange-yellow soluble complex with bismuth. Methods have been developed for the quantitative estimations of copper and cadmium, the separation of copper from cadmium, iron and zinc and for the colorimetric determination of bismuth.

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## Base Exchange, Adsorption, Permeability & Related Properties of Soils

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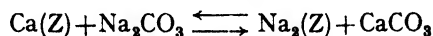
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**Q**UANTITATIVE relationship exists between base exchange capacity (B.E.C.) and the different properties of soils, such as adsorption, exchange reaction, swelling and permeability. The other soil constants affecting these properties are the clay content and the nature of the exchangeable cation.

The following is a summary of the main results obtained by us and is published in the hope that they may be of interest to workers on similar lines elsewhere. The investigation was originally started at the instance of the Irrigation Research Section of the U.P. Government, but now discontinued. The details of experiments and results have been published in the Government Technical Memoranda, which, however, are not easily available to the public. The original problem was to find a suitable lining material for canal beds to prevent losses of water by seepage. The method ultimately recommended involved the treatment of a layer of soils one inch thick with sodium carbonate. A cheaper alternative was found in the substitution of sodium carbonate by a layer of the naturally occur-

ring alkaline soil or *usar*, which, in experimental canals, yielded satisfactory results. Our problem was thus the counterpart of the problem of the reclamation of alkaline soils referred to by Ramdas recently<sup>1</sup>. Both the problems involve the close study of the properties and behaviour of different kinds of soils from practical and theoretical points of view, on which Ramdas and collaborators have already contributed a large number of papers.\*

*Adsorption & Base Exchange* — If a pure calcium soil is treated with a solution of sodium carbonate, the following equilibrium reaction takes place :



This equation does not express all the significant facts connected with the reaction. There is firstly the adsorption of sodium carbonate, and secondly there is

\* The main observations of Ramdas, especially those relating to soils treated with sodium carbonate and calcium chloride are in conformity with our own. The present paper deals only with some of the mathematical relationships which we have investigated.



the base exchange. The two are related but not equivalent. As a rule the sodium carbonate adsorbed is greater than the equivalent of Ca exchanged. Hence the two aspects may be treated separately; the first may be termed general adsorption, while the second leading to the base exchange as exchange adsorption.

A series of adsorption experiments with different strengths of sodium carbonate revealed the fact that the adsorption of sodium carbonate by soils is governed by the well-known Freundlich's adsorption isotherm:

$$y = aC^{1/n}$$

where  $y$  is the quantity of sodium carbonate adsorbed,  $C$  the equilibrium concentration,  $a$  and  $n$ , constants.

A closer study of these two arbitrary constants has shown that " $a$ " is a function of the most important soil constant, the base exchange capacity (B.E.C.). The following table illustrates the idea.

TABLE I

SOIL No.	CLAY %	SILT %	B.E.C. (B)	a	n	a/B <sup>2</sup>
1	52.43	39.20	13.40	0.0497	1.110	$28.0 \times 10^{-3}$
2	27.44	50.25	14.80	0.0721	0.912	$32.6 \times 10^{-3}$
3	16.53	23.08	10.00	0.0352	0.816	$35.2 \times 10^{-3}$
4	6.00	18.50	7.90	0.0186	0.816	$29.0 \times 10^{-3}$
5	27.60	58.90	20.52	0.1350	0.924	$32.0 \times 10^{-3}$
6	26.60	58.90	11.60	0.0404	0.823	$30.0 \times 10^{-3}$
7	24.53	44.24	16.60	0.0854	0.924	$31.0 \times 10^{-3}$
8	21.85	17.36	9.88	0.0303	0.812	$31.0 \times 10^{-3}$
9	26.82	21.24	7.11	0.0160	0.824	$31.7 \times 10^{-3}$

From the last column of the table it is evident that " $a$ " varies as the square of " $B$ ", the base exchange capacity, which is illustrated graphically in Fig. 1, where the relation between " $a$ " and " $B$ " and that between " $a$ " and " $B^2$ " have been plotted. We can now express the Freundlich equation in the form:

$$y = 31 \times 10^{-5} B^2 C^{1/n}$$

in which we have substituted the arbitrary constant " $a$ " by a term involving B.E.C. The value of " $n$ " varies within narrow limits, i.e. between 0.8 and 1.1. Further work is necessary to correlate " $n$ " with other characteristic constants of the soil.

**Exchange Reaction** — It is a peculiarity in surface reactions that adsorption and exchange do not follow the same laws though the two go hand in hand. The adsorption curve is a *parabola*, while the exchange curve is a *hyperbola*. Many attempts have been made to give quantitative expression to the

exchange reaction, and a number of equations proposed by various workers, of which two are:

$$(1) x = \frac{hC}{I + bC} \quad (\text{Langmuir})$$

$$(2) x = \frac{S I}{I + K} \quad (\text{Vageler})$$

where  $x$  = base exchanged,  $C$  = equilibrium concentration,  $I$  = initial concn.,  $h$ ,  $b$ ,  $K$ ,  $S$  constants.

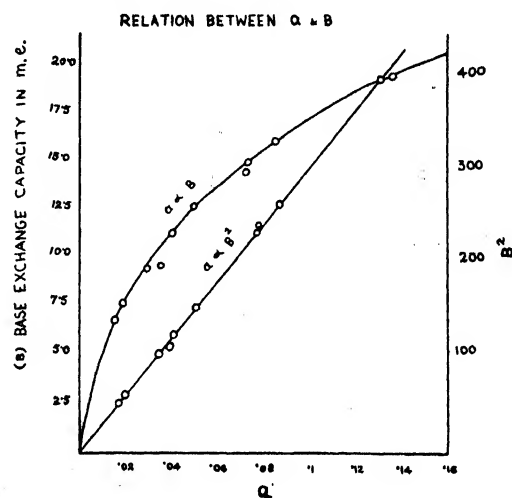


FIG. 1

Both of these equations contain two arbitrary constants. An attempt to replace one of them by the B.E.C. showed the necessity of addition of another constant, but the new equation represented experimental observations more accurately than any other. One typical result is given in Table II.

TABLE II

INITIAL CONCN. Na <sub>2</sub> CO <sub>3</sub> (m.e.)	Ca EXCHANGED OBSERVED (m.e.)	Ca EXCHANGED CALCULATED ACCORDING TO		
		Langmuir	Vageler	Nayer & Shukla
10	0.80	(0.80)	(0.80)	(0.80)
12	0.88	0.867	0.949	0.883
14	0.96	0.953	1.091	0.965
16	1.05	0.992	1.239	1.053
18	1.12	1.000	1.379	1.125
20	1.21	1.158	1.506	1.213
25	1.40	(1.400)	(1.400)	(1.400)

A comparison of the figures in the 2nd and last columns shows that agreement between observed and calculated values is most satisfactory. The merit of the new equation expressed as

$$x = \frac{p + I B}{q + I}$$

(where  $x$  = Ca exchanged,  $I$  = initial concn.,  $B$  = B.E.C., and  $p, q$ , constants) is that it takes into account the most important property of the soil, viz. its B.E.C. Further, Langmuir's and Vageler's equations were found to be not applicable at very low or very high concentrations, while the proposed equation is applicable within wide limits. Thus it is seen that in adsorption and exchange reactions the B.E.C. plays an important rôle, and the course of each reaction can be mathematically predicted.

*An Equation for the Percolation of Water in Ca-Na Soils* — It is well known that sodium soils are generally impermeable to water. When a Ca soil is treated with a sodium salt, exchange takes place between Ca and Na ions, and differing amounts of sodium salt added lead to the formation of different grades of Na-Ca soils. The permeability of such soils containing two such ions can be expressed by the equation (Harris):

$$Y = a \cdot e^{-bS}$$

where  $Y$  = the rate of percolation of water,  $S$  represents the percentage saturation of the soil complex with respect to Na ion, that is,

$$S = \frac{\text{Exch. Na}}{\text{B.E.C.}} \times 100$$

and  $a$  and  $b$ , constants. This equation has been tested in a number of cases and has been found satisfactory. Our object was to find the relation, if any, existing between the arbitrary constants,  $a$  and  $b$ , and the characteristic properties of soils. The following table gives a summary of the results obtained:

TABLE III

SOIL NO.	CLAY CONTENT (C)	B.E.C. (B)	CONSTANT (a)	CONSTANT (b)	a log C	b/B
12	27.44	14.80	0.136	0.0283	0.1967	$1.9 \times 10^{-3}$
2	57.60	20.50	0.136	0.0400	0.1967	$2.0 \times 10^{-3}$
10	26.60	16.60	0.154	0.0320	0.2290	$1.9 \times 10^{-3}$
3	52.43	13.40	0.083	0.0250	0.1430	$1.8 \times 10^{-3}$
13	21.85	9.88	0.178	0.0180	0.2380	$1.8 \times 10^{-3}$
15	26.32	7.11	0.188	0.0130	0.1990	$1.8 \times 10^{-3}$

The significance of the values in the last two columns will be easily understood. The constant " $b$ " is directly related to the B.E.C., while the constant " $a$ " is a function of the clay content. The small variations of values in column 6 might be due to the presence of secondary particles. To clear this point, experiments were carried out with artificial mixtures of the colloidal part of the

soil and pure sand (as an inert constituent) with the following result:

TABLE IV

SOIL NO.	CLAY CONTENT (C)	B.E.C. (B)	(a)	(b)	a log C	b/B
1	100	31.8	0.120	0.057	0.2580	$1.8 \times 10^{-3}$
2	50	15.9	0.148	0.028	0.2500	$1.8 \times 10^{-3}$
3	30	9.54	0.177	0.018	0.2632	$1.9 \times 10^{-3}$
4	25	7.95	0.188	0.016	0.2492	$1.8 \times 10^{-3}$
5	20	6.36	0.195	0.012	0.2542	$1.9 \times 10^{-3}$

"a log C" is thus seen to be definitely a constant and the variation of the values in column 6 of the previous table must be attributed to the presence of secondary particles (size: 0.02-0.002 mm.).

If we now substitute the new values for  $a$  and  $b$  in Harris's equation, we obtain the expression:

$$Y = \frac{k_1}{\log C} e^{-k_2 BS}$$

And since  $S = \frac{(\text{Na})}{B} \times 100$

the final equation is

$$Y = \frac{k_1}{\log C} e^{-k (\text{Na})}$$

where  $k_1$  has the value round about 0.25 and  $k$  is almost exactly equal to 0.18, so that an approximate equation for percolation of water through Na-Ca soils is

$$Y = \frac{0.25}{\log C} e^{-0.18 (\text{Na})}$$

where  $C$  is the clay content, and  $(\text{Na})$  is the exchangeable sodium present in the soil complex. Thus the arbitrary constants in Harris's equation have been replaced by soil constants and numerical constants by an application of ideas underlying B.E.C.

Similar equations have been obtained in the case of soils treated with monovalent and divalent cations, which, however, need not be discussed here.

*The Influence of the Size of the Exchangeable Cation* — The soil permeability, as measured by the rate of percolation of water, seems also to depend on the radius of the exchangeable cation. Experiments carried out with soils saturated with different cations show that under comparable conditions the rate of percolation is expressed by the equation:

$$Y = p \cdot e^{qR}$$

where  $R$  is the radius of the cation,  $p$  and  $q$  being constants. When  $\log Y$  is plotted

against  $R$ , a straight line is obtained for the same series of ions; that is, the monovalent ions Li, Na,  $\text{NH}_4$ , K, Rb all lie in one straight line, while the divalent alkaline earth ions Mg, Ca, Sr, Ba lie in another<sup>8</sup>. This means that the rate of percolation of water through soil colloids saturated with cations is an exponential function of the ionic radius of the exchange cation.

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# Studies in the Constitution of Euphoron — Part I

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**I**N an earlier communication<sup>1</sup> dealing with the characterization of the polycyclic ketone, euphoron, isolated from the dried latex of *Euphorbia tirucalli*, Karimullah and Gopalachari stated that the exact molecular formula for the ketone could not be definitely fixed at that stage of the investigations, as the molecular weight (390 : Rast) and the C, H values of the ketone agreed with the formula  $\text{C}_{27}\text{H}_{42}\text{O}$ , while the iodine value of the ketone and the nitrogen value of its oxime along with their C, H values were found to agree more closely with a  $\text{C}_{29}$  or  $\text{C}_{30}$  formulation. The present communication deals with the results of further studies in the constitution of euphoron. From the evidence now available it is possible to definitely fix on a  $\text{C}_{30}$  formulation for the ketone,  $\text{C}_{30}\text{H}_{48}\text{O}$ . The molecular weight of the ketone (422), determined on the basis of the saponification equivalent of the acetyl ester of the alcohol (euphorol) derived from it, is in good accord with this formula and excludes the alternatives suggested earlier.

The absorption spectrum of euphoron (Fig. 1) shows two bands, one in the region of  $2200\text{\AA}$  ( $E_{1\%}^{1\text{cm.}} = 10$ ) and the other in the region of  $2890\text{\AA}$  ( $E_{1\%}^{1\text{cm.}} = 0.785$ ). There are, however, none in the region of  $2400\text{--}2500\text{\AA}$  which would indicate the absence of any conjugation between either of its two ethylenic linkages and the carbonyl double bond<sup>2</sup>.

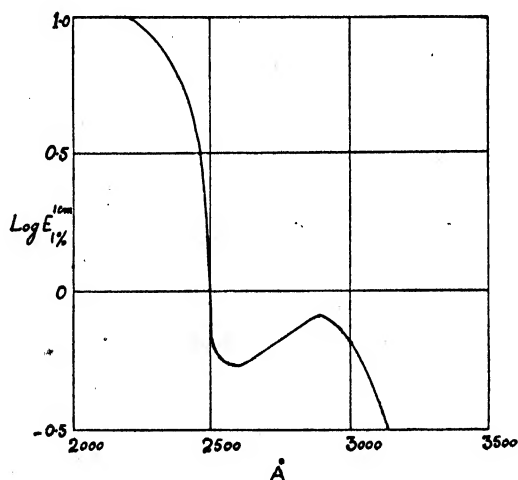


FIG. 1 — ULTRA-VIOLET ABSORPTION SPECTRUM OF EUPHORON.

As reported by the earlier authors, catalytic reduction of euphoron with platinum oxide gave dihydro-euphoron,  $\text{C}_{30}\text{H}_{50}\text{O}$ . Further reduction of dihydro ketone with sodium and moist ether has yielded dihydro-euphorol,  $\text{C}_{30}\text{H}_{52}\text{O}$ . The molecular weight of this alcohol, as derived from the saponification equivalent of its acetate, is in good accord with the formula assigned to it. Oxidation of euphoron with chromic acid in glacial acetic acid medium yielded a ketonic acid which analyses for  $\text{C}_{29}\text{H}_{46}\text{O}_3$  and has been provisionally named euphoronic acid.

As noted by the earlier authors, the use of amyl alcohol and sodium in the reduction of euphoron to the corresponding alcohol euphorol was attended with considerable resinification, which lowered the yield of euphorol and necessitated a tedious repetition of fractional crystallizations for arriving at a pure product. With the use of moist ether in place of amyl alcohol for reduction, it has now been possible to obtain pure euphorol after a couple of crystallizations in a nearly theoretical yield. Euphorol,  $C_{30}H_{50}O$ , has two double bonds like the corresponding ketone and is similarly reduced to a dihydro derivative,  $C_{30}H_{52}O$  (dihydro-euphorol), on catalytic reduction with platinum oxide. Further work on the constitution of these products is in progress.

### Experimental

**Oxidation of Euphoron to Euphoronic Acid**—A solution of chromic acid (2.5 gm.) in 125 c.c. of glacial acetic acid was added drop by drop in the course of 4 hours into a solution of the ketone (5 gm.) in 400 c.c. of glacial acetic acid kept over a boiling water bath. After heating for a further period of 2 hours the solution was concentrated *in vacuo* and then poured into water. The precipitate formed was extracted with ether and the ethereal solution was shaken out with 10 per cent solution of sodium carbonate. The alkaline aqueous layer was neutralized with dil. hydrochloric acid and the precipitate of the crude acid thus obtained was washed, dried and crystallized from methyl alcohol, when euphoronic acid was obtained in the form of colourless needles in a poor yield (0.1 gm.), m.p. 189°-190°. It is soluble in ether, and chloroform in the cold and in methyl and ethyl alcohols in the hot. On crystallization from alcohol, the neutral portion of the oxidation products gave the unreacted ketone (2 gm.) and a viscous liquid (2.5 gm.) from its mother liquor. Found: C, 78.6 per cent; H, 10.6 per cent; neutralization equivalent, 446. Required for  $C_{29}H_{46}O$ : C, 78.7 per cent; H, 10.4 per cent; neutralization equivalent, 442. Required for  $C_{29}H_{48}O$ : C, 78.4 per cent; H, 10.8 per cent; neutralization equivalent, 444.

**Oxime of Euphoronic Acid**—1 gm. of hydroxyl amine hydrochloride was dissolved in 2 c.c. of water and added to 100 c.c. of N/10 alcoholic potash. The solution was filtered from the potassium chloride formed and the filtrate refluxed with 0.1 gm. of euphoronic acid on a water bath. It was then diluted

with water and the precipitate formed was filtered, washed free of alkali, and dried on a porous plate. On crystallization from alcohol, the oxime was obtained in the form of fine, rectangular plates soluble in ether, chloroform, and alcohol, m.p. 214°-215°.

**Euphorol**—A solution of euphoron (5 gm.) in 250 c.c. of ether was taken in a round-bottom flask fitted with a reflux condenser, and 100 c.c. of distilled water was added to it. When the ethereal layer had separated from the lower aqueous layer, 7.5 gm. of sodium were added in small bits in the course of 4 hours. After the completion of the reaction, the ethereal layer was washed free of alkali, dried over anhydrous sodium sulphate, and filtered. On removal of the solvent from the filtrate and crystallization of the nearly colourless crystalline residue from alcohol, pure euphorol, m.p., 108°-109°C., was obtained;  $[\alpha]_D^{25} = +24$ ; found: C, 84.8 per cent; H, 11.9 per cent; M.W., 424; required for  $C_{30}H_{50}O$ : C, 84.4 per cent; H, 11.7 per cent; M.W., 426.

**Euphorol Acetate**—A solution of euphorol in 10 c.c. of dry pyridine was warmed on a water bath and treated with 5 c.c. of acetic anhydride. After half an hour the solution was cooled and diluted with water. The precipitate formed was extracted with ether and the ethereal solution successively shaken out with dil. hydrochloric acid, dil. alkali, and water. After drying over anhydrous sodium sulphate, the solution was filtered and freed from the solvent. On crystallization from alcohol, the nearly colourless crystalline residue gave the acetate in the form of colourless needles, readily soluble in ether and chloroform in the cold and in alcohol and acetone in the hot; m.p. 107°-108°  $[\alpha]_D^{25} = +28.5$ . Found: C, 82.4 per cent; H, 11.3 per cent; saponification equivalent 466, 467, 464. Required for  $C_{32}H_{52}O_2$ : C, 82.1 per cent; H, 11.1 per cent; saponification equivalent, 468.

**Dihydro-euphorol from Euphorol**—A solution of 1 gm. of euphorol was shaken with 0.1 gm. of platinum oxide (Roger-Adams) in a long-necked flask connected to a graduated hydrogen reservoir. The absorption was complete in 24 hours after which the catalyst was filtered off. On concentrating the filtrate, dihydro-euphorol crystallized out in the form of colourless needles, readily soluble in ether and chloroform in the cold and in methyl and ethyl alcohols in the hot; m.p. 122°-123°,  $[\alpha]_D^{25} = +36.0$ .

*Dihydro euphorol from Dihydro-euphoron* — Dihydro-euphoron (5 gm.) dissolved in 250 c.c. of ether was reduced with sodium (7.5 gm.) following the method used for the reduction of euphoron to euphorol. The reduced product was crystallized from alcohol when it gave dihydro-euphorol in the form of colourless needles melting at 122°-123°. When mixed with the dihydro-euphorol obtained from euphorol by catalytic reduction, it showed no depression in m.p. Found : C, 84.1 per cent ; H, 12.3 per cent ; M.W., 431 ; volume of H absorbed, 4.96 lit./100 gm. Required for  $C_{30}H_{52}O$  : C, 84.1 per cent ; H, 12.2 per cent ; M.W., 428 ; volume of H required for one double bond, 5.25 lit./100 gm.

*Dihydro-euphorol Acetate* — Dihydro-euphorol, on treatment with acetic anhydride and dry pyridine, as in the preparation of euphorol acetate, gave the acetate in the form of colourless needles which were readily

soluble in ether, and chloroform in the cold and alcohol in the hot, m.p. 125°-126°,  $[\alpha]_D^{32} = +29$ . On saponification with N/2 alcoholic potash, it gave dihydro-euphorol, m.p. 122°-123°. Found : C, 81.5 per cent ; H, 11.5 per cent ; saponification equivalent, 474. Required for  $C_{32}H_{54}O_2$  : C, 81.7 per cent ; H, 11.5 per cent ; saponification equivalent, 470.

#### Acknowledgement

The authors take this opportunity to express their thanks to Dr. Weiler, Micro-analytical Laboratory, Oxford, for the analytical data and the absorption spectrum curve incorporated in this publication.

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## Some Experiments in Kier Boiling

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THE bleaching of woven cotton goods ordinarily consists of the following three operations, viz. (a) desizing or the removal of the greater part of starch added in sizing ; (b) kier boiling, resulting in the removal of waxy, fatty and other materials such as proteins, pectins, etc. ; and (c) bleaching proper or the destruction of natural colouring matter in the cotton. The skill of the bleacher lies in the efficient removal of the impurities present in the cotton, with the consequent whiteness, with the least damage to the fibres from the point of view of their textile quality. Efficient kier boiling in alkaline liquors is the foundation of successful bleaching. During the kier boiling process, the waxes, oils and fats are in part saponified and in part emulsified, mechanically held dirt is loosened and sizing materials are solubilized. These actions should proceed as nearly as possible to completion. A permanent white with a bril-

liance of tone is ultimately required for the cloth. On the other hand, cloth required for dyeing and printing should be, as far as possible, free from fatty and resinous matter so that it is highly and uniformly absorbent.

The present investigation was undertaken with a view to study the efficiency of a few specially designed kier-boiling treatments in removing the waxy and fatty matter from cotton cloth and yarn so as to render them suitable for the dyeing and printing processes.

#### Materials

*Cloth* — Commercial samples of twill and longcloth made locally from American cotton were used. In one set of experiments, yarn made from short staple Indian cotton was used. The twill in the grey condition contained 2.8 per cent and the longcloth 1.8 per cent of total fatty matter respectively. The yarn contained 0.6 per cent total fatty matter.

The various chemicals used in the boiling and bleaching experiments were commercial products used in the textile trade.

**Colecton B**—This product is marketed by the *Collective Chemical Company Ltd.*, Eagle Road, Bristol, England, as a powerful assistant in laundry washing. It is considered to possess excellent degreasing properties. We are greatly indebted to Dr. K. Venkataraman, Director, Department of Chemical Technology, for making this product available to us for these investigations.

The details of the kier-boiling experiments are given below.

### Experimental Methods

The boiling was carried out in an experimental kier of 3 lb. capacity with a material liquor ratio 1 : 6. The arrangement of the kier and the external heater for liquor is shown in Fig. 1. The cloth was desized as described under each treatment and kier boiled for 6 hr. under 25 lb. steam pressure. The boiled sample was thoroughly washed and bleached with bleach liquor containing 1.5 gm. of available chlorine per litre, and adjusted approximately to a pH of 10.5. At the end of bleaching, the sample was washed and soured using a half per cent hydrochloric acid solution, washed again free of acid, dried and conditioned.

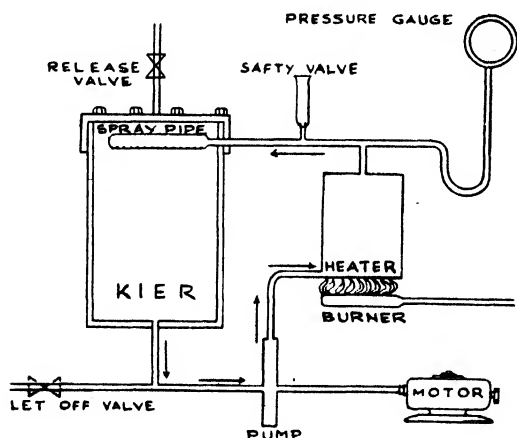


FIG. 1—DIAGRAMMATIC REPRESENTATION OF THE EXPERIMENTAL KIER AND HEATER. The arrows indicate the direction of liquor flow.

### Determination of Total Fatty Matter

The total fat content in the bleached and unbleached samples was determined by extracting a known weight in a Soxhlet apparatus for 8 hr. using a 1:1 mixture of dry benzene and absolute alcohol. The extract

was dried to constant weight at 105°-110°C. and the residual fatty matter expressed on the weight of dry material after correcting for hygroscopic moisture.

### The Kier-boiling Treatment

(1) The grey twill was desized using an enzyme preparation, washed thoroughly and kier boiled using the following 3 recipes : All chemicals are expressed as percentages on weight of cloth.

- (a) 3 per cent caustic soda, 1 per cent soda ash and 0.2 per cent Lissapol C ;
- (b) as in (a) except that Lissapol C was replaced by 0.2 per cent Turkey Red oil ;
- (c) the desized sample was first boiled using 2 per cent lime and 0.2 per cent Lissapol C, washed and again boiled using 3 per cent caustic soda, 1 per cent soda ash and 0.2 per cent Lissapol C.

(2) The twill was desized by grey chemicking with a bleaching powder solution containing 2 gm. of available chlorine for 2 hr. and washed free from chlorine. It was then kier boiled as given below :

- (d) 3 per cent caustic soda ;
- (e) kier-boiled sample from (d) was again boiled in the open with 0.33 per cent Colecton B and 3 per cent soda ash for 2 hr.
- (3) (f) The grey twill was desized as in B and boiled in the open using 0.33 per cent soda ash for 3 hr.
- (4) Grey yarn was kier boiled using
- (g) 3 per cent caustic soda and 0.25 per cent sodium silicate ;
- (h) 3 per cent caustic soda and 0.2 per cent Lissapol C ;
- (i) 3 per cent soda ash and 0.33 per cent Colecton B in the open for 3 hr.

(5) The grey longcloth was desized as in (2) and kier boiled as given below :

- (j) 0.4 per cent Colecton B and 3 per cent soda ash, and
- (k) 3 per cent caustic soda and 0.2 per cent Lissapol C.

### Experimental Results

The results are summarized in Table I.

The saponifiable waxes are normally converted into soaps by the action of alkalis in the kier but the unsaponifiable fatty matter needs special methods for its removal. Of the 4 detergents employed, it is seen that in the case of open soda boil with Colecton B, the saponification of the saponifiable waxes is not complete, but the quantity of total

TABLE I

CLOTH VARIETY	BEFORE BOILING		SAMPLE No.	RESIDUAL FAT % AFTER BOILING	DETERGENT USED
	Total fatty matter % 2.8	Unsaponifiable fatty matter % 0.8			
Grey twill			a	0.61	Lissapol C
			b	0.91	Turkey Red oil
			c	0.53	Two boils with Lissapol C
			d	0.69	nil
			e	0.40	Colecton B
Grey yarn	0.6	0.32	f	0.41	do
			g	0.36	Sodium silicate
			h	0.26	Lissapol C
Grey longcloth	1.9	0.9	i	0.15	Colecton B
			j	0.41	do
			k	0.51	Lissapol C

residual fatty matter in the cloth and the yarn has fallen to a very small value. Scholefield and Ward<sup>1</sup> have shown that under the experimental conditions employed by them using Lissapol A, the wax content of cloth fell to a very small value. In the present investigation, it is seen that the wax content of the cloth even after 2 alkaline boils to which Lissapol C was added, the residual wax content in the boiled cloth is quite appreciable. In view of the observations recorded by Ward and Scholefield, this appears to be surprising.

Turkey Red oil and sodium silicate appear to be the least effective of all in the emulsification of unsaponifiable fatty matter in the

cloth since experiments carried out in absence of any assistant in the kier show much better scouring efficiency (d). Comparing these results with those obtained with Colecton B, it is seen that its emulsifying ability is quite satisfactory. Even by a weaker alkaline treatment of shorter duration, carried out at temperatures lower than those employed in normal kier-boiling processes, the wax content in both the cloth samples and yarn has fallen to a much smaller value.

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## Preliminary Investigations on Brick Clays in India

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A SURVEY of clays used for brick-making in various parts of India has been undertaken by the *Building Research Unit* with a view to ascertain the analytical factors that go to form a good brick. A large number of clay samples were received through the courtesy of provincial governments and states for examination and analysed. The present paper interprets a few of the results obtained.

The brick clays were lightly pounded to break the clods, air dried and sieved through a standard sieve No. 40. Samples passing

through a 2 mm. (No. 10) sieve were used for mechanical analysis by the Bouyouco's hydrometer method, using sodium silicate as deflocculating agent.

The liquid and plastic limits and the plasticity index were determined according to A.S.T.M. D-423 and 424-39 methods<sup>1,2</sup> respectively. The linear and volumetric shrinkages were determined by the methods prescribed by the U.S. Bureau of Public Roads, and the pH by the Beckman glass electrode pH meter in 1:5 soil-water extracts.

The results are presented in Table I.

TABLE I—PROPERTIES OF BRICK CLAYS IN INDIA

SER. No.	LOCALITY	COLOUR	WORK-ABILITY	USER'S OPINION	MECHANICAL COMPOSITION %			LIQUID LIMIT	PLASTICITY INDEX	SHRINKAGE %		pH
					Sand	Silt	Clay			Liner	Volumetric	
1.	Jullundur (Punjab)	Pale yellow	Medium	Good	33	48	19	28	7	4	35	7.5
2.	Mangalore (Madras, W.C.)	do	High	Medium	17	19	64	58	28	13	66	7.0
3.	Dhanbad (Bihar)	Reddish yellow	Medium	Bad	45	28	27	36	12	9	30	6.1
4.	Madras	do	do	Medium	34	32	34	39	20	13	58	7.5
5.	Ootacamund (Madras)	Black	High	Bad	24	26	50	57	23	16	95	8.1
6.	Pipariya (C.P.)	Yellow brown	Low	do	61	22	16	25	9	5	23	7.7
7.	Muzaffarpur (Bihar)	Grey	High	Medium	8	43	54	48	21	7	42	7.8
8.	Chinsura (Bengal)	Ash	do	do	15	44	41	47	23	10	59	7.8
9.	Poona (Bombay)	Red brown	Low	Bad	48	30	22	33	2	4	25	8.0
10.	Sholapur (Bombay)	Brown	do	do	40	30	30	37	13	11	60	8.2
11.	Roorkee (U.P.)	Light yellow	Medium	Good	45	25	30	32	14	6	30	8.2
12.	Delhi	Yellow	do	do	26	52	22	34	18	6	29	8.2
13.	Panchmarhi (C.P.)	Red	Low	Bad	37	28	35	35	14	11	47	8.0
14.	Coimbatore (Madras)	Brown	do	do	45	33	22	62	28	15	88	8.0
15.	Vellore (Madras)	Yellow	High	do	14	46	40	60	26	15	73	7.8
16.	Chittoor (Madras)	Golden yellow	Medium	...	26	35	39	43	17	9	49	8.0
17.	Bhandara (C.P.)	Brown black	do	Bad	50	23	27	36	15	7	32	7.6
18.	Salem (Madras)	do	High	do	14	34	52	56	29	11	87	7.9
19.	Ferozepur (Punjab)	Grey	Medium	Good	19	30	51	35	13	5	29	8.2

### Discussion

The bricks used in India are usually hand-made, and for this purpose the brick clay should be soft and easy to work.

It can be seen from Table I that the clays of the Indo-Gangetic alluvium and those from the west and east coasts of peninsular India generally give good bricks.

The results of mechanical analysis provide an indication of the plasticity of the clays. The sample from Pipariya has a high sand percentage and low plasticity. Muzaffarpur clay has only 3 per cent sand but still gives a poor brick. The calcareous nature of the clay gives the brick a creamy appearance. Samples 9 and 17 show high sand contents and bricks prepared from them are of poor quality.

The liquid limit of a clay indicates its capillary capacity and influences the plasticity index which in turn determines the cohesive property. With increasing plasticity the cohesive characteristics increase, while the permeabilities decrease<sup>4</sup>. The liquid limit is influenced by the clay fraction. In the case of Coimbatore and Vellore samples, the high values for liquid limit suggest that either "active" clay minerals or unusually fine particles are present in the clay fraction; a combination of the two factors<sup>5</sup> may also contribute to the high value. The value of liquid limit for suitable brick clays lies between 35 and 45.

The index of plasticity is greatly influenced by the percentage of sand present in the clay ;

the higher the sand fraction the lower is the index of plasticity. This will be clear from the figures for Poona and Pipariya clays. Materials whose plasticity index lies between 10 and 25 are suitable for brick-making, although for making bricks by hand a value above 25 is considered desirable. The plasticity index for Jullundur clay is 7, and yet the bricks made from it are good. This low value is due to the high silt and low clay content. It has been observed that materials containing equal percentages of clay and silt are easily workable, and are suitable for brick-making.

Volumetric shrinkage is influenced by the silt content of materials. This observation has been corroborated by the work done on soil stabilization, as applied to low-cost housing, carried out in the *Building Research Unit*<sup>6</sup>.

Linear shrinkage is related to the liquid limit of the plastic mass. The higher the liquid limit the higher the linear shrinkage. The range of percentage linear shrinkage considered satisfactory for brick-making is to 5 to 8. Volumetric shrinkage is also related to the liquid limit. The shrinkage is considered high when the value exceeds 60 per cent, medium when it is between 30 and 60, and low when the value is below 30 per cent. Materials which show high values are unsuitable for brick-making—as bricks made from them develop cracks under service conditions. Clays with medium shrinkage values are favoured for brick-making.



The properties of good brick clays are summarized in Table II.

TABLE II

PLACE	MECHANICAL ANALYSIS, %			LIQUID LIMIT	PLASTICITY INDEX	VOLUMETRIC SHRINKAGE %
	Sand	Silt	Clay			
Jullundur	38	48	19	28	7	35
Ferozepur	19	30	51	35	13	29
Roorkee	45	25	30	32	14	30
Musaffarpur	3	43	54	48	21	42
Chinsura	15	44	41	47	23	59
Delhi	26	53	24	34	18	29
Mangalore	17	19	64	58	28	66
Madras	34	32	34	39	20	59

The author's thanks are due to Dr. Gurdas Ram, Officer-in-Charge, Building Research

Unit, Roorkee, for his interest in the work and for many helpful suggestions.

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## Letters to the Editor

### SOLUBILITY AND HEAT OF SOLUTION OF RIBOFLAVIN (VITAMIN B<sub>2</sub>) IN WATER

SENGUPTA AND GUPTA<sup>1</sup> HAVE DETERMINED the solubility of riboflavin at different

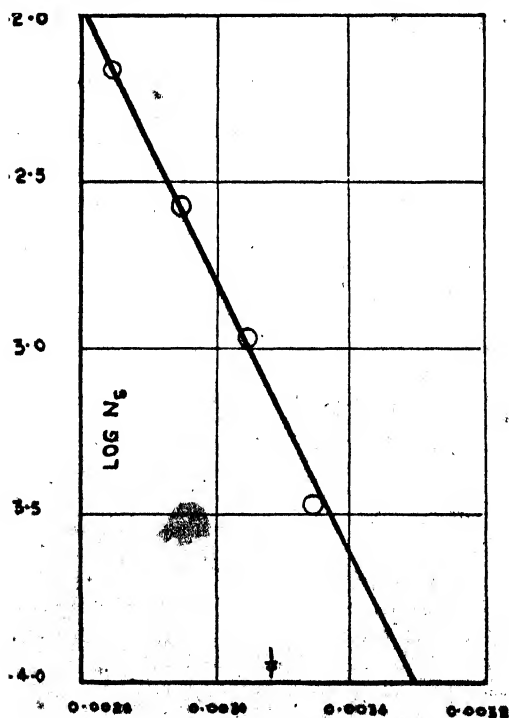


FIG. 1

temperatures, plotted the solubility-temperature curve and extrapolated the solubility curve down to 0°C.

Such direct extrapolation is, however, rendered unreliable by the high curvature of the solubility curve. A better procedure in such cases is to employ the following equation:

$$\frac{d \ln N_s}{dT} = \frac{L_s}{RT^2} \text{ or } \log_{10} N_s = -\frac{1}{2.303} \frac{L_s}{RT} + \text{const.}$$

where  $N_s$  = molar fraction of solute in the saturated solution;

$L_s$  = differential heat of solution at saturation (assumed to be constant over the range of temperature);

$R$  = gas constant; and

$T$  = absolute temperature.

Fig. 1 gives the straight line graph obtained by plotting  $\log_{10} N_s$  versus  $\frac{1}{T}$  which, on extrapolation, gives for solubility of riboflavin at 0°C. the value of 2.9 mg. per 100 c.c. as against 8 mg. per 100 c.c. as shown by the graph of Sengupta and Gupta. From the slope of the curve in Fig. 1, the differential heat of solution of riboflavin at saturation comes to be 9,200 cal. per mole.

A. KALYANASUNDARAM

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June 2, 1949

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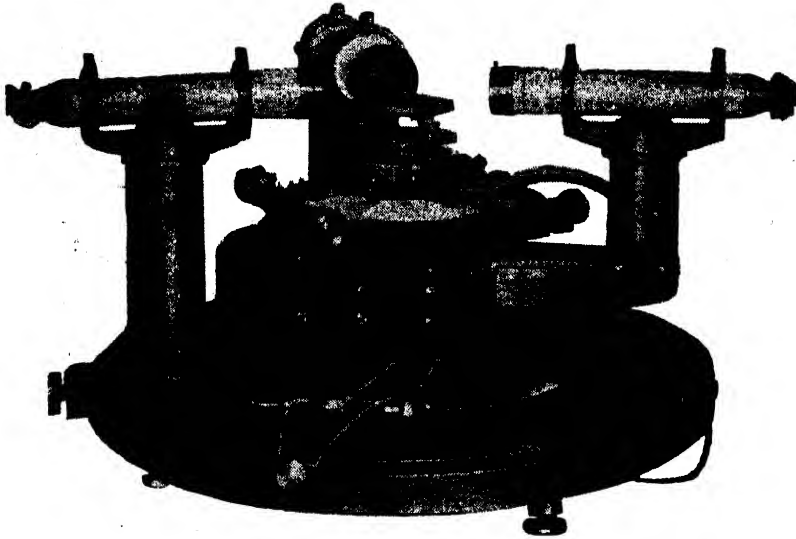
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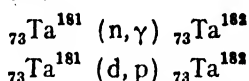
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# Energy Spectrum of Tantalum<sup>182\*</sup>

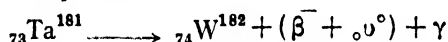
SWAMI JNANANANDA

*The National Physical Laboratory, India, New Delhi*

**T**ANTALUM has a single stable isotope of atomic weight<sup>181</sup> and its abundance is 100 per cent<sup>1</sup>. A radioactive isotope of mass 182 is produced by irradiating this stable isotope with neutrons<sup>2-4</sup> or deuterons<sup>3,5</sup>.



The half life of this radioactive isotope is 97 days<sup>3</sup>. It emits beta and gamma rays, and decays thus:



The beta rays of tantalum<sup>182</sup> were investigated by Rall and Wilkinson<sup>6</sup>, using magnetic spectrograph, and gamma rays by Cork<sup>7</sup>, using magnetic semi-circular focusing spectrograph with photographic registration.

The samples used for the present investigation of beta rays and internal conversion lines of tantalum<sup>182</sup> were obtained by neutron irradiation of tantalum oxide in the Oak Ridge Pile. The finely powdered radioactive tantalum oxide was allowed to fall out of water suspension on to a piece of scotch tape. The spread of the radioactive material, which is uniform, is circular in shape with a diameter of less than 1 mm. The sample was mounted on the source-holder of a magnetic lens focusing beta ray spectrograph, provided with an electrical counter detection and electrically operated mechanical counter. The magnetic lens spectrograph used in this experiment was the one mentioned in a preliminary note published previously<sup>8</sup>. The theory, design and construction of this instrument, however, will be published shortly.

The counting rate of the detecting device was corrected for the decay of the source, the absorption and scattering of the particles by the window of the electrical counter, background of the counter and the magnitude of the counting rate. The corrected counting rate was plotted against the energy in electron volts and the plot is the energy spectrum of tantalum<sup>182</sup>, shown in Fig. 1. The

$\left( \frac{N}{P^2 F_{(Z, E)}} \right)^{\frac{1}{2}}$  values were plotted against energy in MC<sup>2</sup> units, and the plot given in Fig. 2 represents the Fermi analysis of the tantalum<sup>182</sup>.

Fig. 1 shows internal conversion line spectrum, superimposed on a continuous background of tantalum<sup>182</sup>. This beta spectrum is complicated by the presence of a number of distinct peaks, each one representing an internal conversion line. The energy values and the relative intensities of these lines are given in Table I. The present energy values agree with those of the corresponding lines, photographically recorded by Cork<sup>7</sup> who attempted to identify and interpret them as those arising from the transition of tantalum<sup>182</sup> nuclei from excited to ground levels.

The Fermi diagram for the tantalum<sup>182</sup> given in Fig. 2 shows that the spectrum is of an allowed type as indicated by the position of Ta<sup>182</sup> in the Sargent diagram, and that the continuous beta spectrum, besides being complicated by the presence of internal conversion lines, is a composite, consisting of three components, the maximum energy of the hard component being 525.3 Kev. which agrees with the value 521 Kev. obtained from the end point of the natural spectrogram

\* This research was conducted at H. M. Randall Laboratory of Physics, University of Michigan, Ann Arbor, Michigan (U.S.A.).

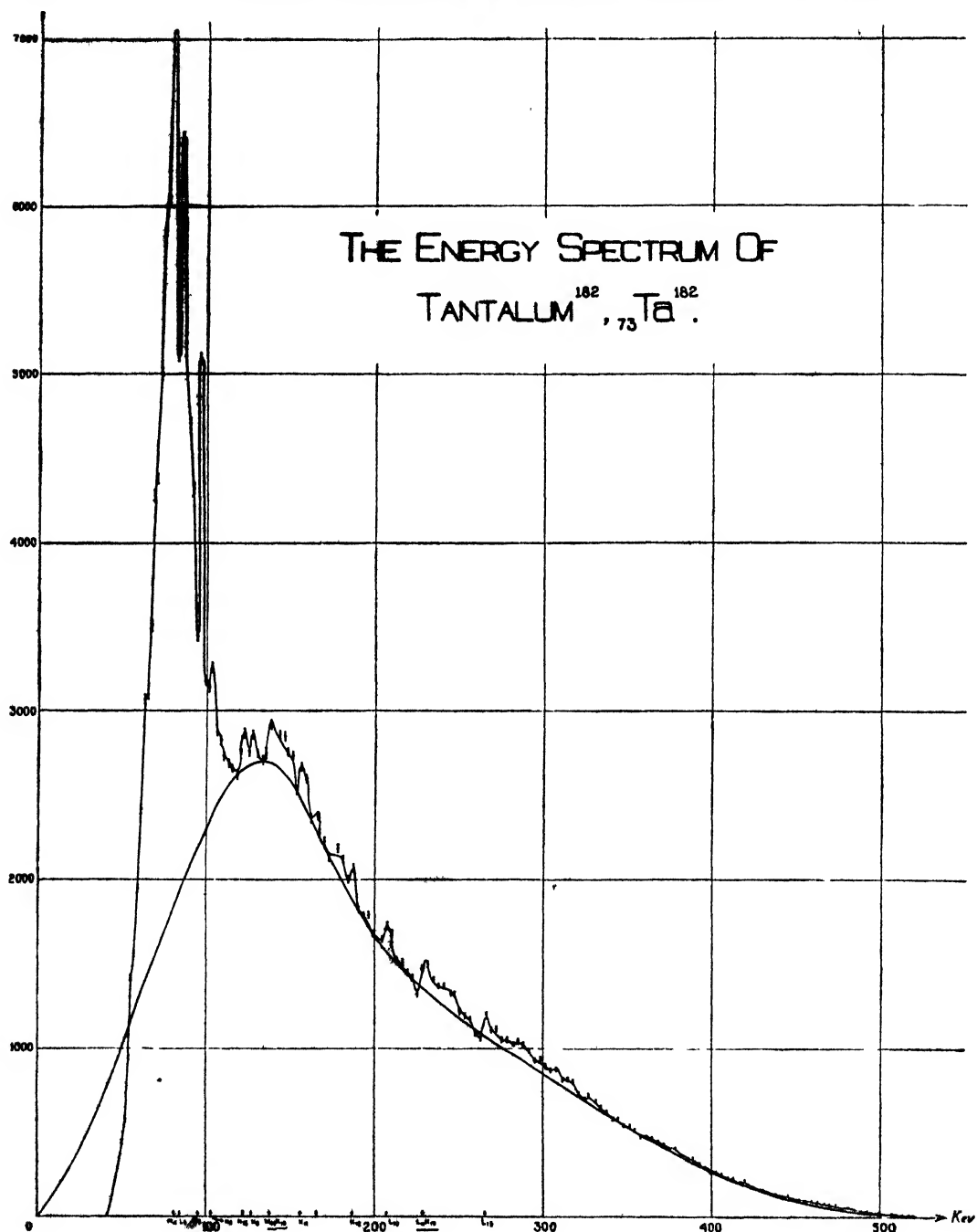


FIG. 1

shown in the Fig. 1. The present energy value of the hard component is in good agreement with the value 530 Kev. obtained by Rall and Wilkinson<sup>6</sup> and also with the value of 0.525 Mev. obtained by Beach, Peacock

and Wilkinson<sup>8</sup>. The maximum energies of the first and the second soft components, as influenced by the hard component, are 457.9 Kev. and 378.1 Kev. respectively. The actual energy values of these soft components

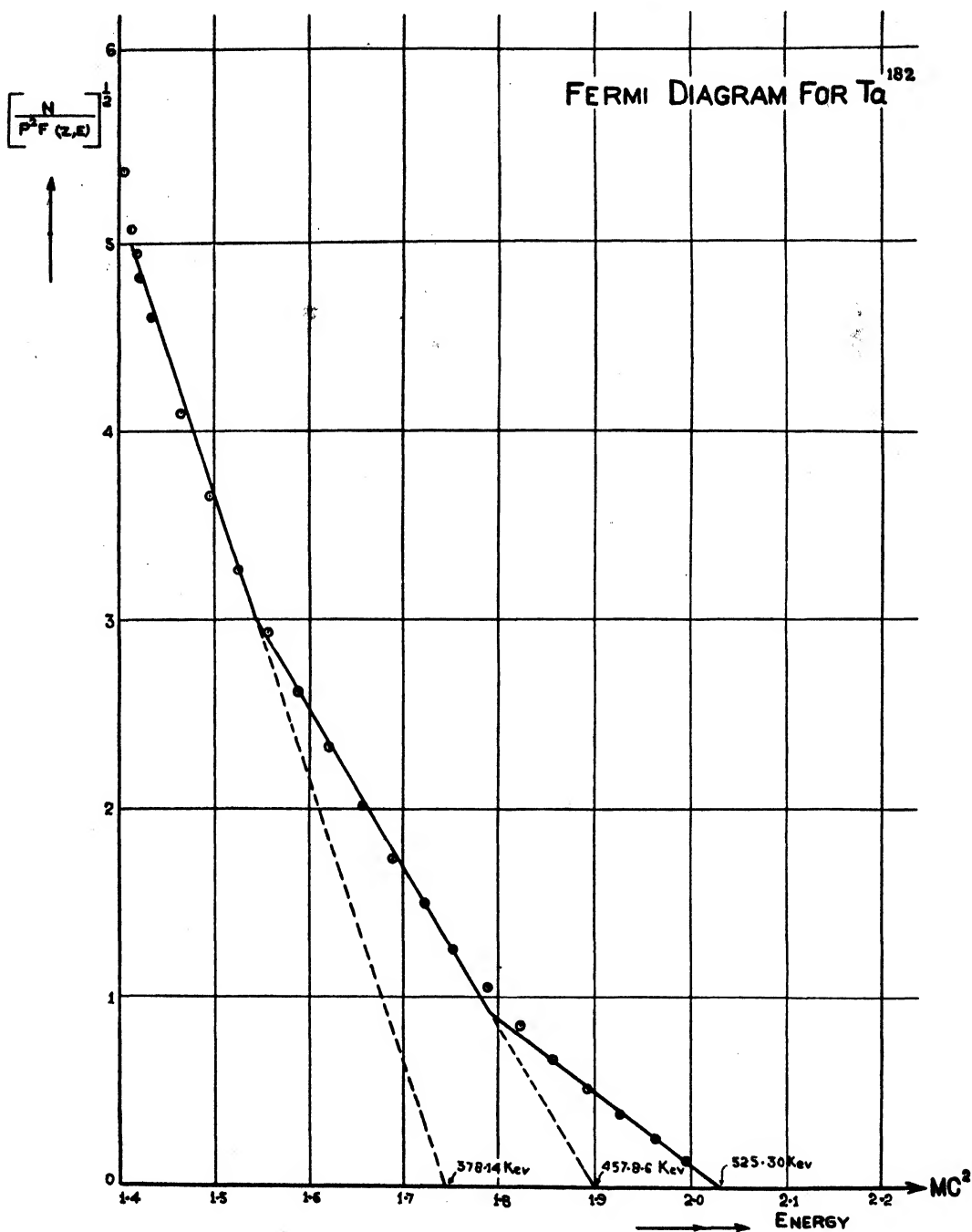


FIG. 2

must, however, be appreciably lower than the values obtained by Fermi analysis of the measured energy values.

Tantalum<sup>182</sup>, as evident from Fig. 2, decays with at least three different beta

components, and, therefore, is likely to transmute into tungsten<sup>182</sup> ground and at least two excited levels, the difference in energy between the ground and higher and lower excited levels being of the order of about

TABLE I

CORK'S IDENTIFICATION & MEASURED ENERGY VALUES OF INTERNAL CONVERSION LINES OF Ta <sup>182</sup>		PRESENT ENERGY VALUES & RELATIVE INTENSITIES OF INTERNAL CONVERSION LINES OF Ta <sup>182</sup>		
Identifica- tion	Energy in Kev.	Energy in Kev.	H $\nu$ in Gauss. cm.	Rel. intensity
M <sub>4</sub>	81.6	80.83	996.4	52.2
L <sub>4</sub>	87.2	85.11	1024.4	44.5
L <sub>3</sub> or K <sub>14</sub>	89.2	...	...	...
M <sub>3</sub>	96.6	96.84	1094.6	28.6
M <sub>2</sub>	98.3	...	...	...
L <sub>7</sub>	100.3	...	...	...
K <sub>8</sub>	107.2	103.24	1136.7	8.8
K <sub>10</sub>	116.6	122.48	1249.0	2.1
K <sub>9</sub>	126.3	127.48	1277.0	1.7
L <sub>14</sub>	131.0	...	...	...
M <sub>14</sub>	138.7	...	...	...
K <sub>10</sub>	148.8	137.69	1333.2	2.2
K <sub>11</sub>	156.9	156.18	1431.4	1.8
L <sub>9</sub>	163.7	...	...	...
M <sub>9</sub> or L <sub>13</sub>	173.6	...	...	...
L <sub>9</sub>	182.4	...	...	...
K <sub>12</sub>	190.0	186.95	1588.8	2.1
L <sub>10</sub>	206.2	207.29	1684.0	1.7
L <sub>11</sub> or K <sub>13</sub>	212.4	231.27	1796.3	2.3
L <sub>13</sub>	245.4	...	...	...
L <sub>10</sub>	271.0	265.63	1950.6	1.6

70 and 150 Kev. respectively. The internal conversion lines due to low energy gamma rays whose energies are of the order of or below 70 and 150 Kev. photographically recorded and interpreted as those arising from Ta<sup>182</sup> by Cork<sup>7</sup> may actually have been those arising from the transition of tungsten<sup>182</sup> nuclei from excited levels to ground level.

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## Chemical Examination of the Drying Oil from the Seeds of *Trichosanthes anguina*, Linn.

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**T**RICHOSANTHES *anguina*, Linn. (English: snake gourd; Hindi: *Chachinda*) belongs to the natural order *Cucurbitaceae*. It is an annual climber cultivated throughout India.

No work has been reported on the seed oil of this plant. The seed fat of *Trichosanthes cucumeroides*<sup>1,2</sup> contains in its component acids about 29 per cent of trichosanic acid while the related *Trichosanthes kadam*<sup>3</sup> has been found to be free from conjugated polyethanoid unsaturation. Since such anomalies are uncommon in related seed fats, it was considered desirable to undertake the chemical examination of the oil from the seeds of *Trichosanthes anguina*, Linn., which belongs to the same genus.

The results obtained are recorded in this paper.

The seeds of *Trichosanthes anguina*, Linn. contain about 32 per cent oil. The oil, when exposed to air, partially polymerizes to a gel in 2 days. When applied on a glass plate with cobalt linoleate as additive, it dries to a non-tacky, soft film in about an hour. But the oil does not set to a jelly-like mass when heated at 293°C. for Browne heat test<sup>4</sup>. This heat-treated oil remains liquid even for a month at ordinary temperature. Such a behaviour has previously been observed by Bradley<sup>5</sup> when tung oil was rapidly heated to 310°C.

The diene value of the oil as determined by the method of Ellis and Jones<sup>6</sup> is 26.4.

Wijs method for the determination of the iodine value is unreliable when conjugated double bonds are present<sup>7,8</sup>, and the total iodine value of the oil (168.4) has been obtained by the Woburn iodine method B. The diene value calculated by subtracting the partial iodine value (Wijs 2 min.) from the total iodine value is much higher (43.1) than that obtained by the direct method of Ellis and Jones. Similar discrepancies have been reported by Cottrell<sup>9</sup>, and have been observed in these laboratories in the case of *kamla* oil<sup>10</sup>.

The oil polymerized to a plastic mass when heated with acetic anhydride, the saponification value of which is identical to that of the original oil.

The separation of the total fatty acids into saturated and unsaturated constituents by the usual lead salt-alcohol method has not been found possible (experimental). Such a behaviour has already been observed in these laboratories in the case of *kamla* oil acids which also contain a major amount of conjugated acid. The diene acid is separated from the total acids by the fractional crystallization of their magnesium salts as adopted by Toyama and Tsuchiya<sup>11</sup> for the fatty acids of balsam pear seed oil. It has been characterized as trichosanic acid. When a petroleum ether solution of the total fatty acids containing a minute quantity of iodine is irradiated by ultra-violet light,  $\beta$ -elaeostearic acid separates out. But contrary to the findings of Kaufmann and Baltes<sup>12</sup> the diene acid could not be completely removed by this treatment. This has also been observed by McKinney and Jamieson<sup>13</sup> in the case of the acids of the American tung oil.

For establishing the presence of oleic and linoleic acids, the residual fatty acids have been oxidized with dilute alkaline potassium permanganate by the method of Sullivan and Bailey<sup>14</sup>, when dihydroxy stearic acid (m.p. 130°-131°C.) and tetra-hydroxy stearic acid (m.p. 153°-155°C.) are obtained. To further prove the presence of linoleic acid, a sample of the residual acids has been brominated by the method of Kaufmann and Baltes<sup>12</sup>. The petroleum ether insoluble linoleic acid tetra-bromide (m.p. 111°-112°C.) has been distinguished from the tetra-bromide of elaeostearic acid by the fact that the material has practically no Wijs iodine value (2.7) even on irradiation.

The proportions of trichosanic, linoleic, oleic and saturated acids in the seed oil of

*Trichosanthes anguina*, Linn. have been calculated from the Rosenmund-Kuhnhenh iodine value, and the diene and thiocyanogen values of the mixed acids, by the equations derived by Kaufmann and Baltes<sup>14</sup> for Chinese wood oil. These results are found to be identical to those calculated by the equations of McKinney and Jamieson<sup>13</sup> for American tung oil. The values for the saturated acids in both cases are similar to that obtained by the Bertram oxidation method<sup>15</sup>.

The saturated acids obtained by the Bertram oxidation method give the neutralization value as 201.7, indicating that palmitic and stearic acids are present to the extent of 22.7 and 77.3 per cent respectively.

The unsaponifiable fraction of the oil consists of sitosterol, a resinous material and a small amount of reddish-brown colouring matter.

#### Experimental

Powdered seeds were extracted 4 times with petroleum ether (40°-60°C.). After removing the solvent, about 32.14 per cent of a dark, reddish-brown oil having the following characteristics were obtained:

Specific gravity at 40°C.	0.9244
Viscosity at 40°C.	58.65 centistokes
Refractive index at 27°C.	1.4963
Acid value	0.343
Saponification value	186.0
Unsaponifiable matter, %	1.42
Acetyl value	Nil
Hehner value	95.31
Soluble fatty acids, %	Nil
Saturated acids (modified Bertram method), %	12.5
Thiocyanogen value	62.5
Hexabromide value	Nil
Iodine value (Wijs)	146.4
Iodine value (Woburn method B)	168.4
Partial iodine value (Wijs 2 min.)	125.3
Diene value (difference of total iodine value and partial iodine value)	43.1
Diene value (maleic anhydride, Ellis and Jones method)	26.4
Browne heat test	Oil does not set to a gel even after heating for 1 hr.

*Isolation & Separation of Free Acids into Solid & Liquid Acids* — 50 gm. of oil were heated on a steam bath for half an hour with alcoholic potash in an atmosphere of nitrogen. The alcohol was distilled off and the soap dissolved in water. The unsaponifiable matter was removed with ether and the soap solution decomposed with dilute



sulphuric acid in an inert atmosphere. The fatty acids were removed by extraction with ether and eventually dried under vacuum at 100°C. They had the following characteristics :

Neutralization value	201.87
Diene value ( Ellis and Jones method )	36.7
Thiocyanogen value	74.05
Iodine value ( Woburn method B )	171.0
Iodine value ( Rosenmund-Kuhnhehn )	124.75
Iodine value ( Wijs )	156.0

30 gm. of the above acids were subjected to Twitchell's lead salt-alcohol process as modified by Hilditch<sup>18</sup>. During recrystallization of the insoluble lead salt from alcohol, a portion of the acids polymerized and remained insoluble in the hot solvent. The amounts of soluble, insoluble and polymerized lead salts, fatty acids and some of their characteristics are given below :

Fatty acids	Amount gm.	Iodine value ( Wijs )	Diene value ( Ellis & Jones )
Soluble lead salt	15.0	144.5	20.2
Insoluble lead salt	8.0	184.5	40.3
Polymerized	6.5	120.7	

*Identification of Trichosanin & Other Unsaturated Acids* — The mixed fatty acids ( 20 gm. ) were dissolved in 120 c.c. of alcohol, neutralized with alcoholic potash ( 5 gm. of potassium hydroxide in 100 c.c. of alcohol ) and 60 c.c. of magnesium acetate solution ( 10 gm. in 100 c.c. of alcohol ) were added. After heating for a short time, the solution was cooled and kept at 10°C. for 18 hr. The insoluble magnesium soaps (A) were filtered, washed with a little alcohol and decomposed with hydrochloric acid. The solid fatty acids liberated were removed by ether. On distilling off the solvent, 14 per cent of yellowish-brown acids having a melting point between 57°-58°C., iodine value 58.2, and neutralization value 202.1 were obtained.

The fatty acids recovered from the filtrate of the magnesium soaps (A) had a neutralization value 202.3 and an iodine value 179.8 ( Woburn method B ). They were again dissolved in 90 per cent alcohol, treated with a solution of magnesium acetate in 90 per cent alcohol, and the precipitate was filtered off. The fatty acids recovered from the filtrate and recrystallized from 80 per cent alcohol yielded trichosanin acid in small laminae. It had the following constants : m.p. 34°-35°C. ; neutralization value

199.7 ; iodine value 261.2 ( Woburn method B ) ;  $d_4^{20}$ , 0.9016 ;  $n_D^{20}$ , 1.5106.

When a solution of the acids in petroleum ether containing a trace of iodine was irradiated by a mercury vapour lamp for 3 hr. and cooled, a large amount of crystalline solid separated. It was filtered and crystallized from alcohol when  $\beta$ -elaeostearic acid ( m.p. 70°-71°C. ) was obtained. The petroleum ether filtrate was concentrated to a small volume and cooled in the refrigerator overnight, which resulted in a further recovery of this acid. This treatment was repeated several times. The residual acids remaining after the removal of the solvent from the final filtrate gave a diene value ( Ellis and Jones ) 8.4. Therefore, it is evident that the mixture still contained appreciable quantities of diene acid.

To 7 gm. of the residual acids, sodium hydroxide ( 8 gm. ) in 800 c.c. of distilled water was added, and the solution were heated for 1 hr. on the steam bath. After cooling, 3.5 litres of ice-cold water were added, then 650 c.c. of 1 per cent potassium permanganate were poured into the soap solution in a thin stream with constant shaking at 10°C. The solution was kept for 8 min. and then decolorized with sulphur dioxide. Concentrated hydrochloric acid ( 160 c.c. ) was added and the liquid allowed to stand for 42 hr. at room temperature. The white precipitate which settled down was filtered and washed with a small amount of petroleum ether. The precipitate was then shaken with dry ethyl ether and afterwards refluxed with ethyl acetate. The combined residue left over after evaporating ethyl ether, and ethyl acetate was crystallized twice from alcohol, when white crystals of dihydroxy stearic acid, melting at 130°-131°C., were obtained, proving the presence of oleic acid.

The undissolved precipitate of the hydroxy acids left over after extraction with ethyl acetate was crystallized from large quantities of boiling water containing a small amount ( 1 c.c. ) of hydrochloric acid. White needles of tetra hydroxystearic acid ( m.p. 153°-155°C. ) were obtained.

To confirm the presence of linoleic acid, the residual acids were dissolved in ether sulphuric, cooled ( -10°C. ) and an ethereal solution of bromine was added in excess with constant shaking. After 2 hours' standing in an ice-salt bath, the excess of bromine was removed with dilute sodium thiosulphate. The solution was evaporated at a low tem-

perature under vacuum. To the dark-brown residue, petroleum ether in excess was added, and the mixture refluxed to a clear solution. After keeping overnight in a refrigerator, colourless needles (m.p. 111°-112°C.) were obtained. The melting point was not lowered when mixed with an authentic specimen of linoleic tetrabromide. To further distinguish it from the tetrabromide of elaeostearic acid, which has the same melting point, the iodine value of this substance was determined (Wij's method) after irradiation with a mercury vapour lamp and was found to be 2.7 (negligible). This showed that the brominated product was tetra bromostearic acid obtained from linoleic acid.

The composition of trichosanic, linoleic, oleic and saturated acids in the seed oil of *Trichosanthes anguina*, Linn. were calculated according to the methods of Kaufmann and Baltes<sup>12</sup> and McKinney and Jamieson<sup>13</sup>. The following results were obtained:

	Kaufmann & Baltes method	McKinney & Jamieson method
	%	%
Trichosanic acid	40.19	55.59
Linoleic acid	15.41	
Oleic acid	25.92	
Saturated acids	12.35	12.32

The saturated acids, as obtained by Bertram oxidation method, were 12.5 per cent. They had a neutralization value 201.7, corresponding to the mean molecular weight 278.1. From these values the composition of the acids can be computed as: palmitic acid 22.7 per cent; stearic acid 77.3 per cent. Various fractions of the acids obtained by crystallization from dilute alcohol gave melting points varying between 60°C. and 66°C.

The composition of the oil, therefore, is the following:

	%
Trichosanic acid	40.19
Linoleic acid	15.41
Oleic acid	25.95
Palmitic acid	2.80
Stearic acid	9.54
Glycerine	4.69
Unsaponifiable matter consisting of sitosterol, a resinous material and a reddish-brown colouring matter	1.42

#### Acknowledgement

The authors record their grateful thanks to Dr. S. Siddiqui for his interest in this investigation.

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# Hypochlorite Oxidation of Cellulose in Presence of Mixtures of Certain Vat Dyes—Part II

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**I**N part I of this communication<sup>1</sup>, the oxidation of cotton cellulose dyed with a mixture of Ciba Blue 2B (oxidizable dye) and Cibanone Orange R (unoxidizable dye) by dilute solutions of sodium hypochlorite at a fixed pH of 9.1, using dyeings prepared from varying concentrations of the dyes, has been studied. The results of these experiments indicated that the powerful accelerating action of reduced Cibanone Orange R was almost completely masked when reduced Ciba Blue 2B was also present on the fibre during hypochlorite treatment. In the absence of Ciba Blue 2B, leuco Cibanone Orange R behaved as a powerful accelerator of the oxidation of cellulose. It was further shown that leuco Ciba Blue 2B did not accelerate the oxidation of cellulose. On the other hand, it itself was attacked by the hypochlorite in preference to the cellulose substrate.

The present investigation gives an account of the experiments carried out, with the systems described in Part I, over a wide range of hydrogen ion concentration of the hypochlorite solutions. Mixed dyeings from the two dyes were prepared as described previously and these were treated with buffered hypochlorite solutions (both in the reduced and unreduced state). The oxygen loss from the solutions was calculated and the oxidized dyeings were examined for their cuprammonium fluidity. The results show that with a decrease in pH of the hypochlorite solution, the influence of Ciba Blue 2B on the accelerating action of Cibanone Orange R becomes less and less pronounced.

## Experimental

The details regarding the dyeing of cotton, the reduction of dyed cotton and the hypochlorite treatment of the leuco dyeings have been given in the earlier paper<sup>1</sup>. The hypochlorite solutions were buffered to suitable hydrogen ion concentration using the following buffers:

Mixtures of M/5 sodium acetate and M/5 acetic acid for pH values from 4 to 5; mixtures of M/15 potassium dihydrogen phosphate and M/15 disodium hydrogen phosphate for pH values from 5.2 to 8; mixtures of M/20 borax and M/5 hydrochloric acid for pH values from 8 to 9.15; and mixtures of M/5 sodium bicarbonate and M/5 sodium carbonate for pH values from 9 to 10.5.

The strength of the hypochlorite solutions was kept as nearly as possible to 3 gm. av. Cl/1. The hypochlorite treated dyeings were examined for their cuprammonium fluidities. These were determined by the method recommended by the B.C.I.R.A.<sup>2</sup>

In Table I are summarized the results obtained when 2 per cent leuco Cibanone Orange R dyeings were treated with hypochlorite solutions. Cibanone Orange R is very resistant to the action of sodium hypochlorite and almost all oxygen lost from the solution should be used up in modifying the cellulose substrate.

TABLE I—TREATMENT OF REDUCED CIBANONE ORANGE R DYEING (2%) WITH DILUTE SODIUM HYPOCHLORITE SOLUTIONS AT 30°C. FOR 10 MIN.

pH OF HYPOCHLORITE SOLUTION	OXYGEN LOST PER 100 GM. OF DYEING TREATED, MILLIMOLES	CUPRAMMONIUM FLUIDITY OF THE TREATED DYEING, 0.5% SOLUTION
4.99	4.5	28.8
5.98	6.8	33.1
6.17	7.8	37.1
7.14	11.5	56.5
7.97	9.0	42.2
8.54	6.2	38.0
9.02	4.3	34.9

In Table II are summarized results obtained when leuco Ciba Blue 2B dyeings were treated with hypochlorite solutions. It is seen that in spite of higher oxygen consumption than in the case of leuco Cibanone Orange R, the cuprammonium fluidities of the treated dyeings are very much lower.

The dyeings appear to be weaker in depth, a considerable amount of the dyestuff being oxidized by the hypochlorite solution.

In Table III the results obtained with the reduced mixed dyeings are summarized. It is seen that the oxygen loss is very much higher than when Cibanone Orange R dyeings were oxidized.

TABLE II—TREATMENT OF REDUCED CIBA BLUE 2B (2%) DYEING WITH DILUTE SODIUM HYPOCHLORITE SOLUTIONS AT 30°C. FOR 10 MIN.

pH OF HYPOCHLORITE SOLUTION	OXYGEN LOST PER 100 GM. OF DYEING TREATED, MILLIMOLES	CUPRAMMONIUM FLUIDITY OF THE TREATED DYEING, 0.5% SOLUTION
4.61	11.4	24.5
5.42	13.9	27.5
5.75	15.9	31.0
6.31	17.2	34.5
7.33	14.2	21.6
8.01	12.4	13.1
9.00	10.3	11.0

TABLE III—TREATMENT OF DYEING PREPARED FROM A MIXTURE OF CIBA BLUE 2B (2%) & CIBANONE ORANGE R (2%) WITH DILUTE SODIUM HYPOCHLORITE SOLUTIONS AT 30°C. FOR 10 MIN.

pH OF HYPOCHLORITE SOLUTION	OXYGEN LOST PER 100 GM. OF DYEING TREATED, MILLIMOLES	CUPRAMMONIUM FLUIDITY OF THE TREATED DYEING, 0.5% SOLUTION
4.51	11.6	31.5
4.76	12.8	33.0
5.59	15.7	33.6
5.80	16.2	37.4
6.09	17.0	43.3
6.33	17.6	46.8
6.95	16.2	35.1
7.90	13.3	21.2
9.02	10.6	13.8

## Discussion

Examination of the results, set out in Tables I-III and shown graphically in Figs. 1 and 2, does not substantiate the conclusions arrived at in part I, viz. that the presence of Ciba Blue 2B completely destroys the accelerating action of Cibanone Orange R. Reference to Fig. 1 shows that the mixed dyeings show approximately the same oxygen consumption as those prepared from Ciba Blue 2B alone. The dyeings prepared from Cibanone Orange R alone, however, consume much less oxygen than either of the above two dyeings treated under identical conditions. If the curves relating the cuprammonium fluidity of the three dyeings to pH of hypochlorite solutions (Fig. 2) are examined, it is seen that in spite of higher oxygen consumption by both the Ciba Blue 2B dyeings and the mixed dyeings, the chemical modification of the cellulose substrate is considerably lower, the curve for the mixed dyeings being midway between

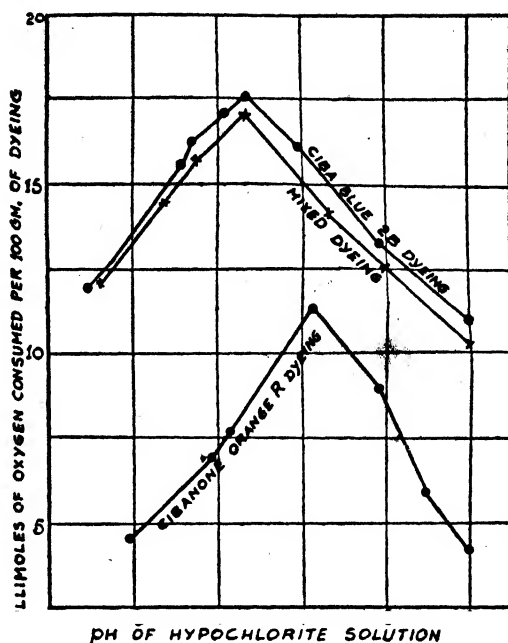


FIG. 1—RELATION BETWEEN OXYGEN CONSUMPTION AND pH OF HYPOCHLORITE SOLUTION.

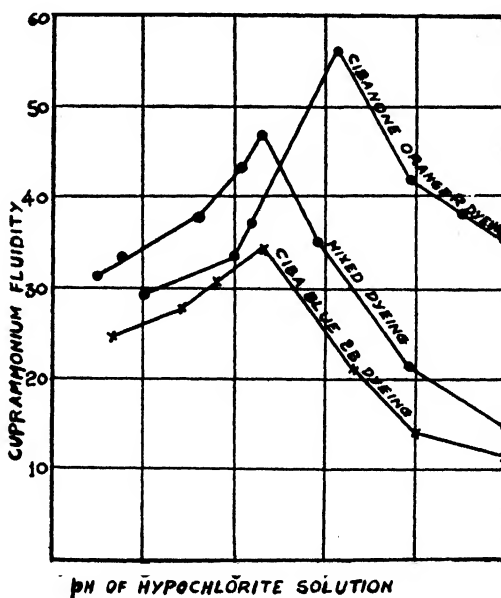


FIG. 2—RELATION BETWEEN CUPRAMMONIUM FLUIDITY OF THE TREATED DYEING AND pH OF HYPOCHLORITE SOLUTION.

the curves for the two dyes, but more or less running parallel to the Ciba Blue 2B curve. This indicates that the Ciba Blue 2B is

oxidized by hypochlorite more readily than the cellulose on which it is dyed.

Another very important point which is clearly brought out is the difference in the  $pH$  for maximum oxygen consumption and maximum cuprammonium fluidity of the oxidized dyeings. Leuco Cibane Orange R dyeings show a maximum oxygen consumption and maximum cuprammonium fluidity at about  $pH$  7.3. Ciba Blue 2B dyeings show the maximum point at about  $pH$  6.3. In the case of the mixed dyeings, the  $pH$  for maximum consumption is the same as that for Ciba Blue 2B and does not appear to be influenced by the presence of Cibane Orange R. Nabar and Turner<sup>3</sup> have shown that when cellulose impregnated with ferrous hydroxide is treated with sodium hypochlorite solutions, maximum oxygen consumption and maximum cuprammonium fluidity are shown to be at about  $pH$  6.3. It has been suggested<sup>4</sup> that the intensity of accelerated oxidation might be conditioned by the reduction potentials of the substance present on the cellulose substrate during oxidation. That this is so is clearly shown by the results of Mhatre and Nabar<sup>5</sup> and Joshi and Nabar<sup>6</sup> who have shown that the intensity of oxidation is dependent on the reduction potentials of ferrous hydroxide and Cibane Orange R respectively. If this is so, the Ciba Blue 2B must, in some way, influence the reduction potentials of Cibane Orange R. This aspect of the work is under investigation and will be discussed elsewhere.

From the foregoing it is clear that the presence of Ciba Blue 2B influences the accelerating action of Cibane Orange R to a considerable extent though its activity is not completely masked as was

believed to be from the results described in part I.

### Summary

1. The oxidation of cellulose, dyed with Ciba Blue 2B; Cibane Orange R and their mixture in the reduced state, by dilute solutions of sodium hypochlorite solutions adjusted to different  $pH$  values, is studied.

2. It is found that reduced Ciba Blue 2B and the reduced mixed dyeings show maximum oxygen consumption and maximum cuprammonium fluidity of the treated dyeings at a  $pH$  of approximately 6.3. The reduced Cibane Orange R dyeings show the maximum point at about  $pH$  7.3.

3. In presence of reduced Ciba Blue 2B, the reduced Cibane Orange R loses its identity as seen from the shift in the  $pH$  for maximum oxygen consumption by the mixed dyeings.

4. The influence of Ciba Blue 2B on the accelerating action of Cibane Orange R on the oxidation of cellulose becomes less and less pronounced with decreasing  $pH$  of the hypochlorite solution.

5. The results indicate that Ciba Blue 2B exerts some influence on the reduction potentials of Cibane Orange R.

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# Influence of Organic Matter on the Bactericidal Efficiency of Indian Essential Oils

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THE germicidal property of essential oils has been known for a long time. Smith<sup>1</sup> investigated the germicidal power of eucalyptus oils. Dyche-Teague<sup>2</sup> and Bryant<sup>3</sup> found that many of the commercial perfumes possessed antiseptic properties. Penfold and Grant<sup>4</sup> and Rideal *et al.*<sup>5</sup> systematically studied the disinfecting properties of Australian essential oils and their constituents. De and Subrahmanyam<sup>6</sup>, De<sup>7</sup>, and Subrahmanyam<sup>8</sup> determined the germicidal values of a number of Indian essential oils and their constituents. Romaine<sup>9</sup>, Reddish<sup>10</sup> and others have studied the disinfecting properties of pine-oil emulsions.

Most of these workers assessed the bactericidal potency against only one test organism, *B. typhosus*, by the Rideal-Walker test in a medium of distilled water. The Rideal-Walker test may not, however, always yield results of direct practical significance. In practice, most often, a disinfectant has to exert its effect in the presence of organic matter in some form or other. A test for practical efficiency of a disinfectant must, therefore, include a suitable form of organic matter in the test medium.

The fact that the germicidal value of emulsified disinfectants is seriously reduced in the presence of faeces has been pointed out by Kenwood and Hewlett<sup>11</sup>. Winter Blyth<sup>12</sup> used both dissolved and suspended organic substances and found a surprising reduction in the activity of coal-tar disinfectants when milk was added to the test mixture. Chick and Martin<sup>13</sup> developed a test by which the disinfectant is tested in the presence of 3 per cent heat-sterilized faeces. Garrod<sup>14</sup> devised a modification of the Chick-Martin test using a 5 per cent suspension of yeast instead of faeces as a standard form of organic matter. Jensen and Jensen<sup>15</sup> determined the phenol coefficients of some disinfectants, after the addition of different quantities of various organic materials by the cover-slip method.

They showed that the phenol coefficient varied with the nature and amount of the organic substance added to the disinfectant. Chick and Martin<sup>16</sup> indicated that adsorption of emulsion particles by some organic substances greatly reduced the germicidal value of the emulsified disinfectant.

In the present investigation, a number of Indian essential oils have been tested for their germicidal efficiency in presence of organic matter. For comparison, parallel determinations of phenol coefficients by the Rideal-Walker test have also been carried out. The germicidal efficiency of the oils has also been measured against different organisms by the Rideal-Walker test.

## Experimental

In the Chick-Martin test<sup>13</sup>, organic matter and disinfectant dilutions are first mixed, and the culture of the test organism added to this mixture after an unspecified interval of time. This is not, however, met with in practical disinfection. In order to bring the test in line with practical conditions, Garrod<sup>14</sup> preferred the procedure of the previous admixture of culture and organic matter. Accordingly, Garrod's modification<sup>14</sup> of the Chick-Martin test has been adopted in our studies. The test is somewhat similar to that of the Rideal-Walker test, the difference being that yeast is present and that the dilutions of disinfectant and phenol are allowed to act on the test-organism for a fixed time of 30 min. instead of varying the periods. In regard to other details, the procedure is the same as that of the Rideal-Walker method.

Dried powdered yeast (5 per cent) is suspended in distilled water. The suspension is autoclaved and subsequently neutralized with sodium hydroxide solution (N/1). For the test, the mixture of culture and yeast suspension is made by adding 1 part of a 24-hr. culture of *B. typhosus* to 24 parts of yeast suspension. 2.5 c.c. of this mixture are added to 2.5 c.c. of dilutions

of the disinfectant and of phenol and kept at a temperature to 18°-20°C. in a thermostat. After 30 min., cultures are made and incubated at 37°C. for 48 hr. The phenol coefficient is calculated by dividing the mean of the highest concentration of phenol permitting growth and the lowest concentration producing sterility, with the corresponding mean of the disinfectant.

For conducting the Rideal-Walker test, every detail, as recommended by Whitby<sup>17</sup>, was rigidly followed. 0.2 c.c. of a 24-hr. culture of *B. typhosus* was exposed to 5 c.c. samples of the different dilutions of the disinfectant and of the phenol for varying periods at a temperature of 18°-20°C. The mixed bacilli and disinfectant were then inoculated into broth tubes at half minute intervals. The inoculated tubes were incubated at 37°C. for 48 hr. and examined for the growth. The Rideal-Walker coefficient was calculated by dividing the figure indicating the degree of dilution of the disinfectant which shows life up to 5 min. but no life thereafter, by that figure indicating the degree of the corresponding dilution of phenol.

Each of the oils, before testing, was freshly steam-distilled, dried over anhydrous sodium sulphate and emulsified under identical conditions by using 1 per cent oil, 1 per cent potassium oleate as the emulsifying agent, and 0.5 per cent potassium carbonate as the emulsion-stabilizer, as suggested by De and Subrahmanyam<sup>8</sup>. The soap and the carbonate were first soaked in water, the oil added and the mixture triturated in a mortar till the oil was properly incorporated and a creamy paste resulted. On dilution to the required volume, the paste formed a milky emulsion which was finally homogenized twice in a small mechanical homogenizer. All the emulsions thus prepared were stable and could be diluted with water in all proportions without any separation of the oil. Potassium oleate used was prepared by refluxing a mixture of alcoholic solution of oleic acid and alcoholic potash in slight excess for about 3 hr. and evaporating off the alcohol and finally drying the soap in a desiccator.

The results obtained are given in Table I.

The results show that of all the oils tested, lemon-grass oil has the highest R-W coefficient as well as the highest phenol coefficient as determined by the Garrod's test, although the latter figure is nearly half that of the former. Attention was primarily directed

TABLE I.—THE EFFECT OF THE PRESENCE OF ORGANIC MATTER UPON THE GERMICIDAL EFFICIENCY OF ESSENTIAL OILS

OIL	R-W. COEFFICIENT	PHENOL COEFFICIENT BY GARROD'S TEST
Lemon-grass oil	17.5	9.2
Cinnamon-bark oil	14.0	7.0
Palmarosa oil	13.0	6.4
Ginger-grass oil	11.5	5.5
Citronella oil	10.0	4.4
Cardamom oil	7.5	3.5
Cubeb oil	6.0	2.8
Fennel oil	6.0	2.2
Clove oil	6.0	2.0
Pine oil	5.5	2.5
Patchouli oil	3.5	1.0
Eucalyptus oil	3.0	1.2
Sandalwood oil	2.0	<1
Turpentine oil	<1	nil

to the study of the germicidal action of lemon-grass oil under various conditions. As in the majority of cases of practical disinfection, different kinds of organic matter are present either singly or in combinations, it seemed of great practical importance to study the influence of various forms of organic matter in suspension or in a solution, as the case may be, upon the bactericidal efficiency of lemon-grass oil emulsion. Organic materials such as faeces, milk, gelatin, starch, etc., were used. Garrod's<sup>14</sup> modification of the Chick-Martin test was followed using one of these organic materials instead of yeast. For comparison, similar tests on phenol solution were also carried out. The phenol coefficient was determined after contact with both phenol solution and phenol solution with addition of organic material. The results obtained are given in Table II.

Of the several oils tested, those which gave fairly high R-W coefficients against *B. typhosus* were selected and their bactericidal efficiencies were assessed against several Gram-positive and Gram-negative organisms, as also an acid-fast organism. Among the Gram-negative, most of the organisms under *Coli*, *Typhoid*, *Salmonella* and *Dysentery* groups, and among the Gram-positive, *Pneumococcus* and a few organisms under *Streptococcus* and *Staphylococcus* groups were employed as test organisms. All the organisms grew readily on the R-W broth with the exception of only *Streptococcus haemolyticus* and *Pneumococcus*. For the growth of these two organisms, serum broth was used. For testing against *Vibrio cholerae*, the pH of the R-W broth was adjusted at 8.0 instead of the usual pH of 7.6. *Mycobacterium phlei* grew in the R-W broth after 72 hr. of incubation at 37°C. The results obtained are tabulated in Table III.



**TABLE II—GERMICIDAL POWER OF LEMON-GRASS OIL R.W. COEFF. 17.5) IN PRESENCE OF VARIOUS ORGANIC SUBSTANCES**

ORGANIC SUBSTANCE	CONCENTRATION %	PHENOL COEFFICIENT (GARROD) CALCULATED WITH	
		Phenol	Phenol + organic substance
Faeces	3	6.4	6.9
Faeces	6	3.2	4.1
Urine	3	10.5	10.6
Urine	6	8.3	8.5
Faeces	3		
+ Urine	3	6.1	6.6
Sewage water	3		
Sewage water	6	9.5	9.6
Serum	3	7.6	7.9
Serum	6	6.3	6.8
Saliva	3	3.0	3.8
Saliva	6	15.2	15.2
Milk	3	13.3	13.3
Milk	6	5.5	5.9
Egg albumin	3	3.0	4.3
Gelatin	3	9.1	9.3
Starch	3	9.0	9.2
Gelatin	3	11.3	11.5
+ Starch	3		
Peptone	3	7.8	8.1
	3	12.5	12.6

NOTE: The various organic materials used for the tests were obtained under sterile conditions. The albumin was withdrawn from fresh eggs (hen's) under aseptic conditions and mixed with three parts of sterile physiological salt solution. Urine was filtered through a Chamberland filter. Pasteurized milk was used. The faeces sample obtained from a human subject was dried at 100°C., powdered and passed through a fine sieve. The suspension of the fine particles in distilled water was autoclaved and subsequently neutralized with alkali.

**TABLE III—GERMICIDAL EFFICIENCY OF ESSENTIAL OILS AGAINST DIFFERENT ORGANISMS**

ORGANISM	R-W COEFFICIENT				
	Lemon-grass oil	Cinnamon-bark oil	Palmarosa oil	Ginger-grass oil	Citronella oil
<b>Gram-negative</b>					
<i>B. typhosus</i>	17.5	14.0	13.0	11.5	10
<i>B. typhosus A</i>	18.5	14.5	14.0	12.0	10
<i>B. typhosus B</i>	18.5	14.5	14.0	12.0	10
<i>B. typhosus C</i>	18.5	14.5	14.0	12.0	10
<i>B. enteritidis</i>	17.5	14.5	13.5	11.0	9
<i>B. shigae</i>	16.0	13.0	13.0	9.0	9
<i>B. flexneri</i>	16.0	12.0	13.0	9.0	9
<i>B. coli</i>	18.5	12.0	14.0	11.0	10
<i>B. aerogenes</i>	18.5	12.0	14.0	11.0	10
<i>Proteus vulgaris</i>	15.0	14.0	12.0	7.0	8
<i>Vibrio cholerae</i>	12.5	8.0	8.5	5.0	5
<i>B. pyocyaneus</i>	14.0	9.0	10.0	8.0	7
<b>Gram-positive</b>					
<i>Staphylococcus aureus</i>	5.5	2.0	4.0	2.0	2
<i>Staphylococcus albus</i>	5.5	4.0	4.0	3.0	3
<i>Streptococcus pyogenes</i>	5.5	2.0	3.0	2.0	2
<i>Streptococcus haemolyticus</i>	4.0	1.0	2.0	1.0	1
<i>Pneumococcus</i>	2.0	1.0	1.0	<1.0	<1
<b>Acid-fast</b>					
<i>Mycobacterium phlei</i>	<1.0	<1.0	<1.0	<1.0	<1

## Discussion

It is obvious from Table I that the disinfecting power of the emulsions prepared out of essential oils is reduced to a large

extent in presence of yeast suspension. The phenol coefficients obtained in a medium of distilled water by the R-W test are in most cases more than double the values obtained by the Garrod's test. The latter values, however, give a more correct indication of the disinfecting power of the oils from the point of practical disinfection. Of all the oils tested, lemon-grass oil has the highest bactericidal action as demonstrated in the medium of distilled water as well as in the presence of organic matter. The other oils which also possess high germicidal action are cinnamon-bark oil, palmarosa oil, ginger-grass oil and citronella oil.

From Table II it is evident that the disinfecting power of phenol is affected but slightly by the addition of various organic substances, whereas that of the emulsified disinfectant is reduced to a considerable extent. The interference varies with the nature and condition of the organic matter added. The suspended matter appears to reduce the efficiency of the disinfectant to a more pronounced extent than the dissolved one. In presence of 3 per cent faeces, serum or milk, the disinfecting power of lemon-grass oil is reduced to about one-third of its original R-W coefficient.

The results given in Table III show that in comparison with the 5 oils tested, lemon-grass oil gives the highest R-W coefficient as determined against different organisms. In general, the essential oils are found to be highly active against Gram-negative organisms. The activity against Gram-positive organisms, however, is very low and practically no bactericidal action is shown when tested against the acid-fast organism.

It has thus been demonstrated that of all the essential oils examined, Indian lemon-grass oil possesses the highest bactericidal action against different pathogenic organisms in a medium of distilled water as well as against *B. typhosus* in the presence of different kinds of organic matter. Hence, further systematic work was taken up with a view to attempting to standardize all the conditions necessary for the preparation of a suitable disinfectant out of lemon-grass oil and the results will be reported in subsequent communications.

## Summary

1. Several Indian essential oils were emulsified and tested for their bactericidal efficiency both by the Rideal-Walker method in a medium of distilled water, and by



Garrod's modified method of the Chick-Martin test in presence of a 5 per cent yeast suspension. The germicidal action of each oil was found to be considerably reduced in the presence of yeast suspension.

2. Lemon-grass oil, having been found to be most active, was subjected to further systematic tests in the presence of different kinds of organic matter. Notable reduction in germicidal activity was observed in the presence of faeces, serum and milk.

3. The bactericidal efficiency of the oils which gave high R-W coefficients against *B-typhosus* was further measured against different organisms and was found to be generally high against Gram-negative bacteria but low against Gram-positive organisms. The activity against the acid-fast organism was practically nil.

#### Acknowledgement

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## Relationship Between the Chemical Constitution of the Constituents of Certain Essential Oils & Their Bactericidal Properties

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THE relationship between the chemical constitution of terpenes and their germicidal action has received very little attention. De<sup>1</sup> attempted to correlate the disinfecting properties with the chemical constitution of different terpene alcohols, aldehydes, ketones, phenols, oxides, esters, hydrocarbons and sesquiterpenes, and indicated that the compounds containing —OH, —CHO, —CO— and —O— groups were generally active. No quantitative study on the comparative influence of these groups on the germicidal power has, however, been carried out so far.

The high bactericidal properties of several Indian essential oils, viz. lemon-grass, cinamon-bark, palmarosa, ginger-grass and citronella oils have been reported in a

previous communication<sup>2</sup>. In the present study, the chief constituents from these essential oils have been isolated and examined for their germicidal activity. Further, in order to demonstrate the influence of the chemical structure of these constituents on their bactericidal power, compounds have been prepared from them and tested for germicidal action. The extent to which the presence of the —OH and —CHO groups in the terpene alcohols and aldehydes enhance germicidal efficiency with special reference to the degree of unsaturation as well as the position of the double bond has been clearly shown.

#### Experimental

Citral was isolated<sup>3</sup> from lemon-grass oil by mechanically shaking the oil with a

solution of a mixture of sodium sulphite and sodium bicarbonate and finally liberating citral from the solution of the addition compound formed by alkali and ether.

Cinnamic aldehyde was isolated<sup>4</sup> in a similar way from cinnamon-bark oil using a saturated solution of sodium bisulphite.

Citronellal was isolated from Ceylon citronella oil by the method suggested by Boulez<sup>5</sup> using a solution of a mixture of sodium bisulphite and sulphite.

Geraniol was isolated<sup>6</sup> from palmarosa oil by forming the compound of geraniol with freshly fused powdered calcium chloride and finally decomposing it with water.

Geranic acid was prepared<sup>7</sup> from citral by forming first citral-oxime with hydroxylamine hydrochloride, then converting the oxime into the nitrile of geranic acid with acetic anhydride and finally by hydrolysing the nitrile with alcoholic potash.

Citronellic acid was prepared<sup>8</sup> in a similar way from citronellal.

Citronellol was prepared by reducing citronellal by sodium and anhydrous ethyl alcohol by the usual method of reduction.

Hydro-cinnamic aldehyde was prepared by oxidizing hydro-cinnamyl alcohol (phenyl propyl alcohol) with a mixture of potassium dichromate and sulphuric acid by the standard method of oxidation.

The methyl ethers of geraniol, citronellol, cinnamyl alcohol and hydro-cinnamyl alcohol were prepared<sup>9</sup> by forming first the chloride of the corresponding alcohol using thionyl chloride and anhydrous pyridine and subsequently by converting the chloride formed with methyl alcohol and sodium.

Each of the compounds was vacuum-distilled at 10 mm. pressure. The refractive

index was determined by the Abbe refractometer at 20°C. The other compounds were secured from the *B.D.H.*

As mentioned in the previous communication<sup>2</sup>, the various compounds were emulsified under identical conditions by using 1 per cent of substance to be tested, 1 per cent potassium oleate as the emulsifying agent and 0.5 per cent potassium carbonate as the emulsion-stabilizer, and examined for the Rideal-Walker coefficient using *B. typhosus* as the test organism. In a few cases where the substance was solid, it was first dissolved in the least possible amount of alcohol and then emulsified. The results are given in Table I.

### Discussion

The comparative figures for the bactericidal properties of terpene aldehydes and their corresponding alcohols, viz. citral and geraniol, citronellal and citronellol, cinnamic aldehyde and cinnamyl alcohol, hydro-cinnamic aldehyde and hydro-cinnamyl alcohol, show that in general the —CHO group is more powerful than the —OH group.

The extent of influence of the presence of the aldehydic group in citral, cinnamic aldehyde, citronellal and hydro-cinnamic aldehyde on their bactericidal efficiency is demonstrated from the most pronounced reduction in the R-W coefficients of the corresponding acids, viz. geranic, cinnamic, citronellic and hydro-cinnamic acids, the last 2 acids being devoid of any germicidal activity.

The importance of the hydroxyl group for maintaining the germicidal potency of the terpene alcohols is obvious from a comparison of the R-W coefficients of the alcohols, viz.

TABLE I — RELATIONSHIP BETWEEN CHEMICAL CONSTITUTION & R-W COEFFICIENT

COMPOUND	CHEMICAL FORMULA	CONSTANTS		R-W COEFFICIENT
		B. p. at 10 mm., °C.	Ref. index at 20 °C.	
Citral	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{C}(\text{CH}_3) : \text{CH}.\text{CHO}$	107-109	1.4873	20.0
Citronellal	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{CH}(\text{CH}_3).\text{CH}_2.\text{CHO}$	86-88	1.4572	12.5
Cinnamic aldehyde	$\text{C}_6\text{H}_5.\text{CH} : \text{CH}.\text{CHO}$	118-120	1.6180	16.5
Hydro-cinnamic aldehyde	$\text{C}_6\text{H}_5.\text{CH}_2.\text{CH}_2.\text{CHO}$	102-105	1.5228	8.0
Geraniol	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{C}(\text{CH}_3) : \text{CH}.\text{CH}_2.\text{OH}$	111-114	1.4768	15.5
Citronellol	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{CH}(\text{CH}_3).\text{CH}_2.\text{CH}_2.\text{OH}$	98-100	1.4558	8.0
Cinnamyl alcohol	$\text{C}_6\text{H}_5.\text{CH} : \text{CH}.\text{CH}_2.\text{OH}$			11.0
Hydro-cinnamyl alcohol	$\text{C}_6\text{H}_5.\text{CH}_2.\text{CH}_2.\text{CH}_2.\text{OH}$			5.0
Geranic acid	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{C}(\text{CH}_3) : \text{CH}.\text{COOH}$	149-152	1.4860	2.5
Citronellic acid	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{CH}(\text{CH}_3).\text{CH}_2.\text{COOH}$	141-143	1.4542	<1
Cinnamic acid	$\text{C}_6\text{H}_5.\text{CH} : \text{CH}.\text{COOH}$			2.5
Hydro-cinnamic acid	$\text{C}_6\text{H}_5.\text{CH}_2.\text{CH}_2.\text{COOH}$			<1
Methyl geraniol ether	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{C}(\text{CH}_3) : \text{CH}.\text{CH}_2.\text{O}.\text{CH}_3$	87-88	1.4688	4.5
Methyl citronellol ether	$(\text{CH}_3)_2\text{C} : \text{CH}.\text{CH}_2.\text{CH}_2.\text{CH}(\text{CH}_3).\text{CH}_2.\text{CH}_2.\text{O}.\text{CH}_3$	79-82	1.4541	1.0
Methyl cinnamyl ether	$\text{C}_6\text{H}_5.\text{CH} : \text{CH}.\text{CH}_2.\text{O}.\text{CH}_3$	100-103	1.5734	3.0
Methyl hydro-cinnamyl ether	$\text{C}_6\text{H}_5.\text{CH}_2.\text{CH}_2.\text{CH}_2.\text{O}.\text{CH}_3$	88-92	1.5318	1.0

geraniol, citronellol, cinnamyl and hydro-cinnamyl alcohols, with those of the corresponding methyl ethers. The R-W coefficient of the methyl ether is remarkably reduced as compared with that of the corresponding alcohol.

Again, in spite of the presence of the  $-CHO$  group, the bactericidal efficiency of citronellal is much lower than that of citral; also, the efficiency of hydro-cinnamic aldehyde is significantly lower than that of cinnamic aldehyde. Similarly, the bactericidal efficiencies of citronellol and hydro-cinnamyl alcohol are appreciably lower than those of geraniol and cinnamyl alcohol respectively, although the hydroxyl group is present in each of the 4 compounds. These results show that unsaturation in general and, especially, the presence of the  $\alpha$ - $\beta$  double bond enhances the germicidal activity of terpene aldehydes and alcohols.

The slight activity of geranic and cinnamic acids and methyl ethers of geraniol and cinnamyl alcohols may be ascribed to their unsaturation owing to the presence of the double bond in the  $\alpha$ - $\beta$  position.

### Summary

1. Some of the important constituents of the Indian essential oils were isolated and examined for their R-W coefficients.

2. The bactericidal action of the compounds has been correlated with their chemi-

cal constitution. The  $-CHO$  group has been found to be generally more active than the  $-OH$  group. The presence of an  $\alpha$ - $\beta$  double bond in terpene aldehydes and alcohols enhances the efficiency of the active group.

3. The extent of influence of aldehydic and alcoholic groups on the bactericidal action has been demonstrated. The acids prepared from aldehydes and the methyl ethers prepared out of alcohols have been found to be practically devoid of germicidal activity.

### Acknowledgement

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## Preliminary Investigations on the Physico-chemical Properties of Some Indian Bricks

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A LARGE variety of bricks are used in India for building purposes. They have distinctive characteristics of their own depending on the properties of the clays from which they have been made and the methods employed in their manufacture. Data on the properties and characteristics of the various brick types in use in the country is not available

and the present investigation relates to a study of some brick types in use.

### Experimental

Samples of bricks were obtained from 20 different localities (TABLE I) and are representative of the types manufactured from the clays available in the respective localities.

TABLE I

SAMPLE No.	LOCALITY	BURNING CHARACTERISTICS	COLOUR	SOUND	SHAPE	STRUCTURE & COMPOSITION	RESISTANCE TO	
							Breaking	Abrasion
1	Rupar HCC ( Punjab )	Well burnt	Dull red	Metallic	Very good	Even ( in some cases there is layer or lump formation )	Hard	Hard
2	Amritsar AS ( Punjab )	do	do	do	do	Even ( lump formation and iron nodules )	do	do
3	Jullundur KLM ( Punjab )	do	Brick red	do	do	Even ( air holes )	do	do
4	Delhi M45	do	Dull, brick red	do	do	Uneven ( air holes )	do	do
5A	Roorkee PMW ( U.P. )	Under burnt	Yellowish red	Dull	Fair	Even	do	Medium
5B	Roorkee SJS ( U.P. )	Well burnt	Copper	Metallic	Good	Even ( containing mica flakes )	do	Hard
6	Muzaffarpur APD ( Bihar )	do	Yellow skin up to 0.5 cm., buff core	do	Very good	Even	do	do
7	Dhanbad SBS ( Bihar )	do	Dull red	Dull	Good	Uneven ( air holes, mica flakes, quartz crystals, lumps of iron nodules, carboniferous matter )	Easy	Medium
8	Chinsurah ROSE ( Bengal )	do	Crimson red	Metallic	Fair	Even ( air holes )	Hard	Hard
9	Bhandara BHS; NH ( C.P. )	Under burnt	Dark red	Dull	do	Uneven	Easy	Soft
10	Pachmarhi DK ( C.P. )	Well burnt	Pale, brick red	do	Good	Uneven ( flint grains )	Very easy	do
11	Piparia ( C.P. )	do	Dark orange	do	Fair	Even ( air holes )	Not very hard	do
12	Belgaum ( Bombay )	Under burnt	Yellowish red	do	Good	Fairly even ( air holes, iron nodules )	do	Medium
13	Dowladshwaram VBK ( Madras )	do	Copper	Semi-metallic	Fair	Uneven ( air pockets due to burnt straw, flint, stones, etc. )	Easy	Soft
14	Chepauk CS ( Madras )	Well burnt	Brick red	do	Good	Even ( flint and iron nodules )	Very hard	Hard
15	Mettur ( Madras )	Under burnt	Light chocolate	Dull	do	Uneven ( air holes and stones )	Very easy	Soft
16	Vellore ( Madras )	Well burnt	Brownish red	Semi-metallic	do	Even ( air holes and stones )	Easy	do
17	Ootacamund JRK ( Madras )	do	Chocolate red	Metallic	Fair	Uneven ( air holes, stones, burnt straw )	Hard	do
18	Coimbatore ( Madras )	do	Dull red	Semi-metallic	do	Even ( air holes )	Easy	do
19	Anantpur ( Madras )	do	Pale chocolate	do	Good	Even ( air holes, stones )	do	do
20	Calicut CTL ( Madras )	do	Bright red	Metallic	Very good	Even ( a few burnt iron nodules )	Not very hard	Hard

The bricks were tested for the following : (i) bulk density; (ii) percentage porosity; (iii) compressive strength; (iv) pressure deficiency; (v) permeability; (vi) rate of water absorption; and (vii) chemical composition.

The bulk density of the brick was calculated by cutting out a cube from the brick and determining its dimensions and weight.

The porosity and compressive strength were determined according to standards laid down in B.S.S. No. 1257 (1945). The porosity was determined by boiling a brick in water for 5 hr. and allowing it to cool overnight; the difference in weights before and after this treatment gave the porosity. The compressive strength was determined by crushing the brick in a hydraulic press between 2 thin pieces of plywood after the frogs have been filled up, and the bricks planed by 1 : 1½ cement mortar. A study of the rate of absorption of water by bricks immersed in water was also made.

Permeability was determined by allowing water to pass through the brick under a known pressure head and measuring the amount of water passing through it per unit time.

To determine the pressure deficiency, 2 methods were employed. In one method<sup>1</sup>, a piece of brick was waxed round the sides and sealed in a funnel-shaped apparatus. The apparatus was filled with air-free water and as evaporation took place on the free and unwaxed surface of the brick, water in the tapering end of the apparatus was drawn towards the surface. This caused a rise in the mercury column of the manometer attached to the apparatus. The pressure registered by the manometer is a measure of the capillary force. This method is slow, and the testing takes a long time. In the second method<sup>2</sup>, suction, just sufficient to overcome the capillary force of the brick, is applied as in the method originally used

for the determination of pressure deficiency of sand and silt. The method is rapid and has been adopted for determining the pressure deficiency of bricks also. The values obtained by this method are invariably higher than those obtained by the first method.

The results of these tests are given in Table II. The rate of absorption of water is represented graphically in Fig. 1.

### Discussion

Some of the accepted characteristics of a good brick are that it should be of a good shape, hard to scratch, have a metallic ring when struck, and be free from foreign matter. It was, however, noticed that most of the bricks studied contained stones and carboniferous matter. These inclusions weakened the bond between the different brick particles. This can be avoided by sieving the clay before moulding. The lumps in the brick are responsible for low compression strength values even though the bricks were good in other respects.

A study of compressive strengths of bricks (TABLE II) shows that 5 samples have a value of 1,000-2,000 lb./sq. in. while 7 others have a strength of 500-750 lb./sq. in., and the rest below 500 lb./sq. in., the lowest being 157 lb./sq. in. for a sample from Pachmarhi

(C.P.). Most of the bricks tested are weak, except those made from the alluvial soils of the Indo-Gangetic tract. Considering the importance of bricks as building material, it is important to study the mechanism by which a brick derives its strength and to devise methods by means of which bricks of high compressive strength can be obtained from different types of Indian soils.

The mechanical strength of a brick depends upon: (i) the internal resistance of the particles constituting the brick; and (ii) the bond provided by the vitrification of the clay. The higher the firing temperature, the better is the vitrification and, hence, the stronger is the bond. Soils are made up of particles of different sizes. Particles greater than 0.02 mm. diam. are classified as sand, those between 0.02 mm. and 0.002 mm. as silt, and those less than 0.002 mm. as clay. It is the sand fraction that provides the greatest internal resistance. Consequently, a certain percentage of sand is essential for getting strong bricks. The chemical composition of the bricks (TABLE III) shows that bricks whose strength is between 1,000-2,000 lb./sq. in. contain 70-80 per cent of silica. However, there are samples containing silica within the above range but are still weak. Thus, samples from Piparia, Mettur and Bhandara

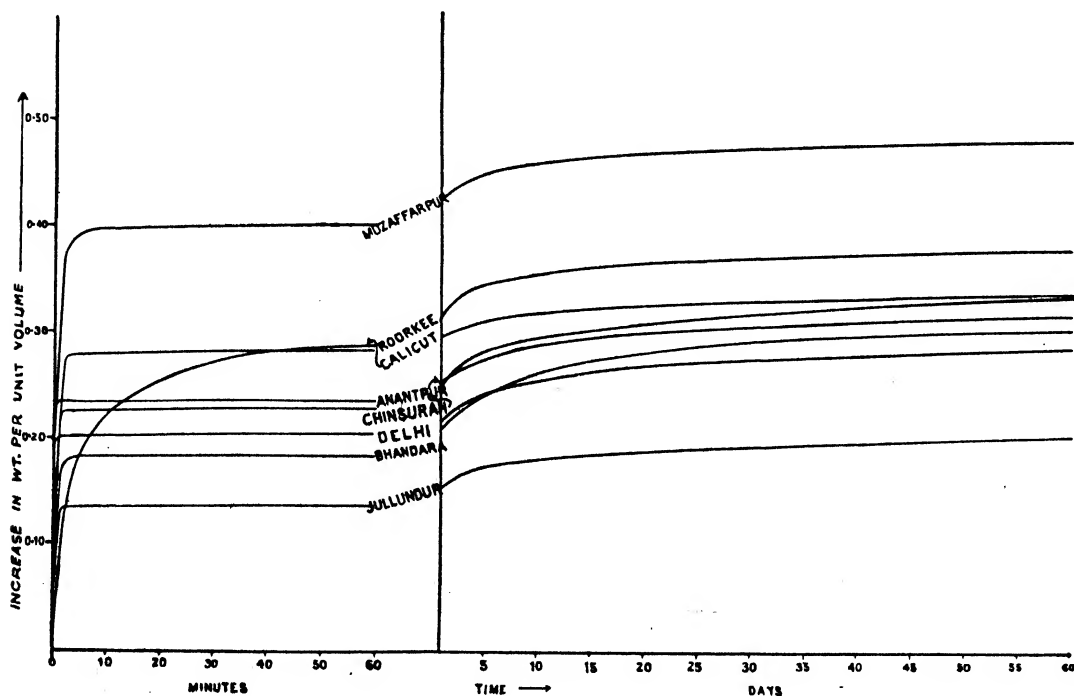


FIG. 1

TABLE II

SAMPLE No.	ACTUAL DENSITY OF BRICK POWDER gm./c.c.	BULK DENSITY OF BRICK gm./c.c.	POROSITY, % OF WT. OF SAMPLE	COMPRESSIVE STRENGTH lb./sq. in.	INCREASE IN WT. ON SOAKING IN WATER FOR 24 HR. %	ABSORPTION OF WATER IN 1 HR. ; % OF WATER ABSORBED IN 24 HR.	PRESSURE DEFICIENCY		PERMEABILITY $\times 10^{18}$
							Evaporation method, cm. of water	Suction method, cm. of water	
1	2.85	1.65	35.8	1792	16.8	92.2	...	637	...
2	2.57	1.74	32.1	1165	14.0	90.3	...	406	...
3	2.43	1.87	23.0	1142	8.2	91.4	...	460	...
4	2.60	1.77	33.7	1993	12.8	94.0	206	349	1.90
5A	2.56	1.65	37.6	1254	19.4	90.7	182	...	0.12
5B	2.58	1.74	33.1	1725	15.4	89.9	240	...	1.80
6	2.54	1.33	47.4	492	31.8	94.8	371	...	0.16
7	2.46	1.58	36.0	425	17.0	92.3	55	123	8.80
8	2.52	1.71	32.0	694	15.6	91.2	483	871	0.55
9	2.56	1.72	32.8	470	12.9	87.2	176	579	0.98
10	2.37	1.68	30.0	157	15.3	93.5	36	173	3.30
11	2.44	1.72	29.6	649	12.2	90.3	115	358	1.50
12	2.68	1.62	39.7	739	19.2	92.5	...	513	...
13	2.44	1.47	41.0	582	23.6	85.8	64	...	3.20
14	2.56	1.80	29.0	358	13.4	84.6	97	443	2.35
15	2.50	1.70	33.9	179	13.4	80.2	59	167	4.00
16	2.60	1.72	33.8	582	11.3	95.4	75	348	2.40
17	2.81	1.67	40.6	515	18.1	91.7	36	86	15.00
18	2.54	1.57	38.0	515	18.5	95.7	...	175	...
19	2.43	1.58	35.0	358	16.5	96.7	...	143	...
20	2.58	1.64	36.5	627	18.8	97.0	196	795	...

TABLE III

SER. No.	SILICA $\text{SiO}_2$	SESQUIOXIDES $\text{R}_2\text{O}_3$	LIME $\text{CaO}$	MAGNESIA $\text{MgO}$	ALKALI SALTS AS $\text{NaCl}$ & $\text{KCl}$	SULPHATE AS $\text{SO}_3$	LOSS ON IGNITION
1	77.37	15.50	2.80	1.90	3.38	0.03	0.28
2	74.86	19.62	1.75	0.74	3.42	0.10	0.25
3	75.46	19.86	3.02	0.63	1.91	0.05	0.25
4	79.61	15.07	2.14	1.70	1.50	0.01	0.21
5	71.75	23.43	2.01	1.15	1.05	0.03	0.26
6	51.43	19.99	24.71	0.46	2.60	0.01	2.13
7	80.54	15.59	1.16	0.41	3.80	0.05	0.29
8	69.42	23.66	3.52	1.51	2.29	0.04	0.43
9	71.00	22.31	1.95	1.28	0.72	0.01	1.55
10	63.76	27.33	1.48	1.23	1.90	0.01	3.83
11	79.00	15.68	1.94	2.57	1.51	0.01	0.80
12	71.75	21.64	3.11	0.74	0.50	0.02	0.90
13	69.99	24.01	3.94	1.70	1.21	0.03	0.40
14	70.52	22.79	2.60	0.04	3.31	0.05	0.31
15	72.31	19.95	3.41	1.34	1.40	0.02	0.40
16	59.69	20.01	6.01	0.56	3.76	0.02	0.66
17	52.91	36.42	5.80	2.36	4.25	0.07	0.23
18	63.22	20.21	5.97	0.71	2.07	0.03	0.44
19	73.80	18.65	3.79	0.41	3.90	0.06	0.38
20	55.32	36.86	2.88	0.99	0.81	0.01	2.15

contain 79 per cent, 72 per cent and 71 per cent silica respectively, yet they are weak. This may be due to the fact that the firing temperature for these bricks was low or the soil cannot give good bricks at the temperatures attainable in the ordinary brick kiln. Results of the mechanical analysis of the soil from which bricks are made are given in Table IV.<sup>3</sup> Though the analysis does not represent the results on actual soil samples from which the bricks under test were made, yet it gives a fairly good idea of the sand content of the soils from which these bricks were obtained. It is seen that the sand content of Piparia and Bhandara soils are as high as 61 per cent and 50 per cent while those of Delhi and Jullundur are only 26 per cent and 33 per cent respectively. This clearly indicates that the bulk of silica in Piparia and Bhandara bricks is derived from the coarser fraction, while in the case

TABLE IV

SAMPLE No.	MECHANICAL COMPOSITION		TOTAL SILICA CONTENT %	ACTIVE SILICA %	INACTIVE SILICA %
	Coarse fraction (sand) %	Fine fraction (silt & clay) %			
1	...	...	77	...	...
2	...	...	75	...	...
3	33	67	76	43	33
4	26	74	80	54	26
5	45	55	72	27	45
6	3	97	51	48	3
7	45	55	80	35	45
8	48	52	69	21	48
9	50	50	71	21	50
10	37	63	64	27	37
11	61	39	79	18	61
12	...	...	72	...	...
13	...	...	70	...	...
14	34	66	70	36	34
15	...	...	72	...	...
16	14	86	60	46	14
17	24	76	53	29	24
18	45	55	63	18	45
19	...	...	74	...	...
20	17	83	55	38	17

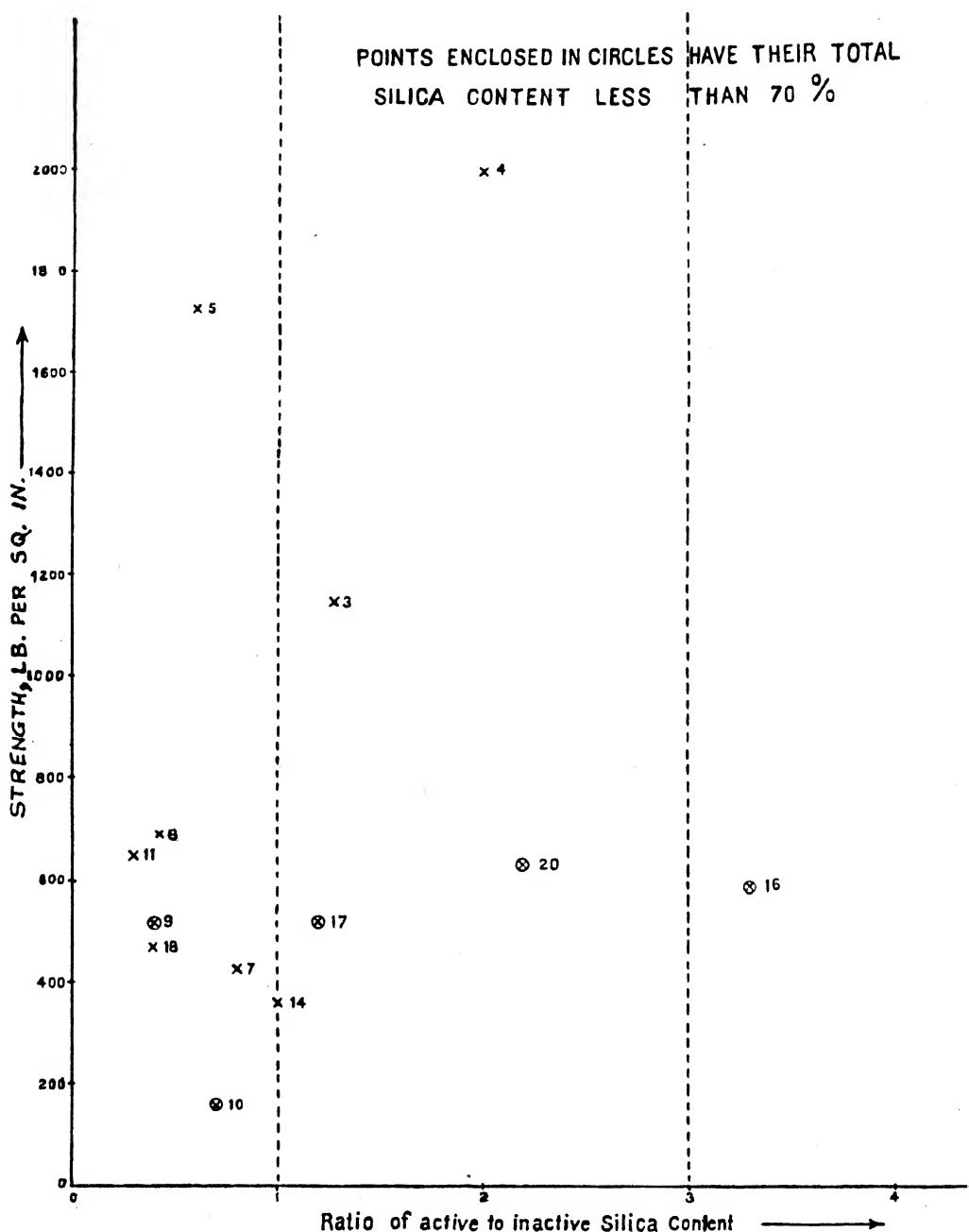


FIG. 2

of Delhi and Jullundur, it is from the finer fraction. Silica from the finer fraction is supposed to be more active due to a greater surface exposed for chemical reaction than the silica derived from the coarser fraction. Thus, for the present discussion, silica from finer fraction is termed *active silica*,

while that from the coarser fraction is termed *inactive silica*. Active silica helps in the vitrification of the brick, while inactive silica provides greater internal frictional resistance than the active silica. It seems that (FIG. 2) there is a definite ratio of active to inactive silica (between 1 and 3)

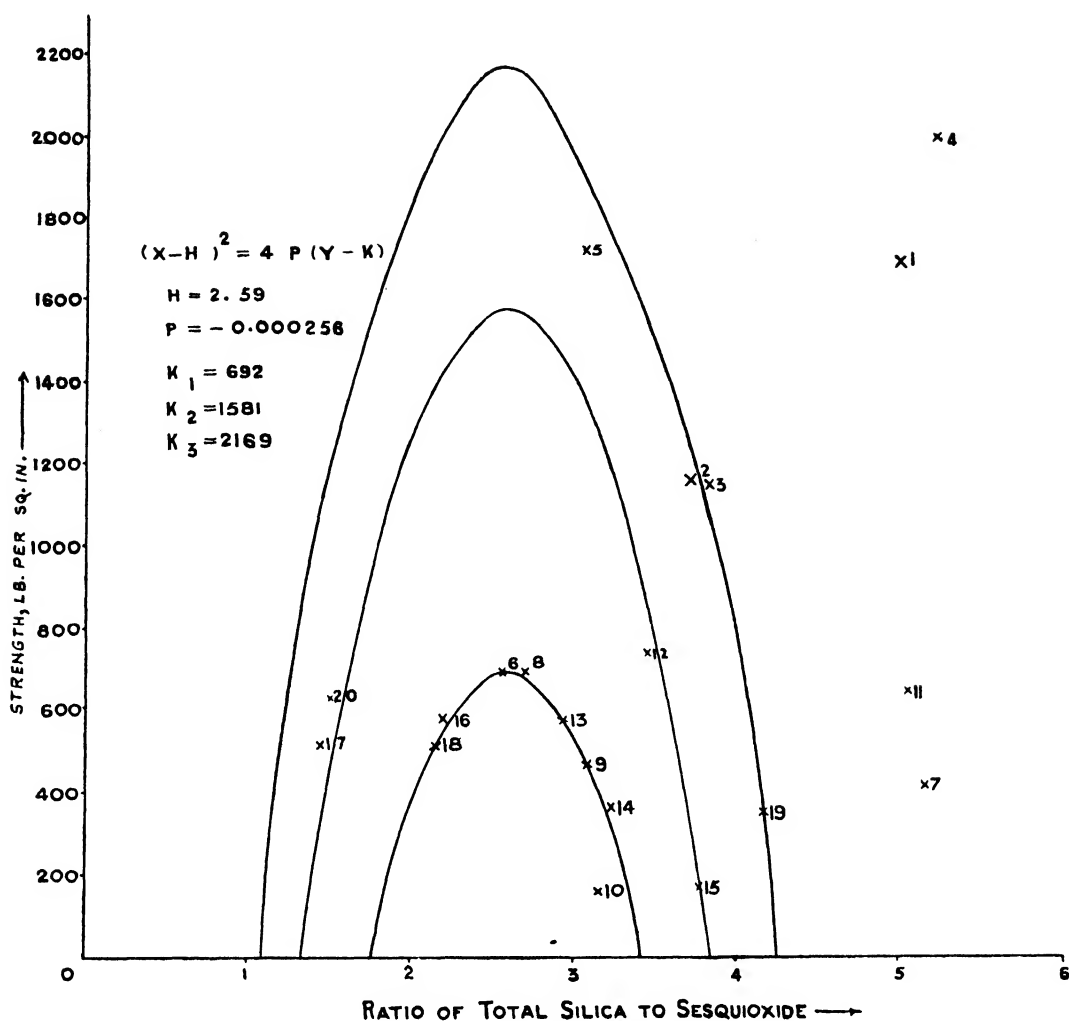


FIG. 3

at which a brick of high compressive strength is obtained. If this view is correct, then samples from Muzaffarpur, Vellore and Calicut should improve in strength by an admixture with a certain quantity of coarse sand, while those from Bhandara, Pachmarhi, Piparia and Coimbatore should improve by an addition of fine sand. It may be pointed out here that these results are not conclusive, but provide guidance for further work on the subject.

If the compressive strength of the bricks are plotted against the ratio of silica to sesquioxide contents of the bricks (FIG. 3), the graphs obtained are a series of parabolas having their foci lying on the same vertical axis and having the same value of the para-

meter  $p$  in the equation  $(x-h)^2 = 4p(y-k)$ ,  $(h, k)$  being the vertex of the parabola. This shows that there is only one definite ratio of silica to sesquioxide contents which gives the maximum strength. It is not yet clear what factor or factors are responsible for this result. Probably firing temperature is one of the factors.

Water absorption and saturation coefficients of a brick sample are useful guides in gauging the durability of a brick. The water absorption values of the brick samples are given in Table II. The rates of absorption of water are about the same in all bricks. It is found that they absorb the major part in the first 2 minutes and thereafter it is slow. In this respect,



the 24-hr. absorption test, as described in B.S.S. No. 1257 (1945), can be usefully shortened to 1 hr. period as the bricks absorb, in the first hour, 92 per cent of the water absorbed in 24 hr. period. A great deal of time can be saved if this test is modified for the 1 hr. period. The 5-hr. boiling test can also be similarly shortened as it is found that boiling for 1 hr. suffices to bring about the same absorption as boiling for 5 hr. does within 1 per cent error.

The subject of pressure deficiency has been discussed in detail in a previous paper<sup>4</sup>. Results of pressure deficiency (TABLE II) show that weaker bricks have smaller pressure deficiencies.

#### Acknowledgements

Thanks are due to officers of the P.W.Ds. of various provinces in India for supplying brick clays, bricks and other materials. Thanks are also due to the authorities of the Thomason College of Engineering, Roorkee, for providing testing facilities.

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## A New Method for the Analysis of Adulterated Graphite

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NATURAL graphite, when marketed in the form of fine powder, is often adulterated with carbonaceous materials like charcoal, coke and coal, which cannot be detected easily. Spence<sup>1</sup> has described a few qualitative tests to identify them but no method is available to estimate the graphite content in the adulterated mixture. The usual method of analysis of graphite by combustion is not suitable.

A possible means of estimating graphite content in an adulterated mixture is by burning away the adulterants at as low a temperature as possible, so chosen that loss of graphite by oxidation would be minimum. The ignition temperature of graphite in oxygen, according to Moissan, is 690°C., although oxidation starts at a lower temperature. The ignition temperature of charcoal and coke varies widely, being dependent upon the temperature at which they were prepared. However, it varies from 345°C. for charcoal<sup>2</sup> to 600°C. for coke<sup>3</sup>, both being lower than that of graphite.

An attempt was made to burn off the adulterants by heating the sample in a

porcelain boat in a tube furnace, maintained at a temperature below 600°C., but as the progress in combustion could not be watched, it resulted in the combustion of a good amount of graphite. It was also found that the temperature of the mixture should be varied, a higher temperature being required at the start to ignite the adulterant. Once combustion starts, the temperature could be lowered, because enough heat is liberated by combustion of the adulterant to maintain the mass hot. Constant stirring is essential to expose a fresh surface of the adulterants for rapid combustion. To maintain these conditions, a quantity of the mixture was heated in a porcelain or silica crucible with its lid off, over a small bunsen burner, with constant stirring.

#### Experimental

Mixtures of finely ground (—150 mesh) crystalline and amorphous graphite with charcoal, coal and coke, were prepared from materials of known composition.

About 1 gm. of the dried sample was weighed into a tared porcelain crucible

TABLE I

Ceylon graphite: volatile matter = 1.70%; ash = 2.00%;  
C = 96.30%; ash in charcoal = 17.11%; coal = 17.78%;  
coke = 17.74%

EXPT. No.	ADUL- TERANT	LOSS DUE TO ADUL- TERANT % (a)	ASH % (b)	GRAPHITE % 100 - (a + b)	GRAPHITE % CALCU- LATED
1	Charcoal	45.53	10.53	43.94	42.65
2		45.83	10.53	43.65	42.65
3		32.32	7.63	60.05	61.53
4		31.96	7.63	60.41	61.53
5		19.34	5.23	75.43	77.09
6	Coal	18.68	5.23	76.09	77.09
7		39.74	9.69	50.57	50.22
8		39.42	9.69	50.89	50.22
9		24.96	6.60	68.44	69.50
10		24.51	6.60	68.89	69.50
11	Coke	19.60	5.87	74.53	73.98
12		20.43	5.87	73.70	73.98
13		44.43	11.24	44.33	45.05
14		43.35	11.24	45.41	45.05
15		29.23	8.30	62.48	63.04
16		28.86	8.30	62.84	63.04
17		20.30	6.58	73.12	75.18
18		19.97	6.58	73.45	75.18

TABLE II

Mysore graphite: volatile matter = 2.52%; ash = 33.00%;  
C = 64.48%; ash in charcoal = 6.17%; coke = 17.74%

EXPT. No.	ADUL- TERANT	LOSS DUE TO ADUL- TERANT % (a)	ASH % (b)	GRAPHITE % 100 - (a + b)	GRAPHITE % CALCU- LATED
1	Charcoal	22.53	27.02	50.45	51.83
2		21.67	27.02	51.31	51.83
3		46.77	19.72	33.51	32.56
4		47.25	19.72	33.03	32.56
5		28.03	24.93	47.04	48.39
6	Coke	27.64	24.93	47.43	48.39
7		29.87	27.71	42.42	42.11
8		30.54	27.71	41.75	42.11
9		18.85	29.72	51.43	50.65
10		19.34	29.72	50.94	50.65
11		5.84	31.70	62.46	61.32
12		7.30	31.70	61.00	61.32

(45 c.c. capacity). The lid was removed and the crucible heated on a small bunsen burner (temperature about 700°C.). The temperature of the contents of the crucible was slowly raised till particles of the adulterant began to glow. The temperature was maintained as low as possible, consistent with efficient burning of the adulterant. The contents of the crucible were constantly stirred with a stout nichrome wire.

Complete combustion of the adulterant takes 5 to 20 min. depending on its nature and quantity, more time being necessary for coke and less for charcoal. When the glowing ceased, the temperature was slowly raised with constant stirring to ensure complete combustion of the adulterant. This "end point" in the experiments could be easily determined in the case of charcoal and coal, but in the case of coke, it is rather difficult. However, with practice, the difference in colour of the red-hot graphite, which is rather dull, and coke could be judged and the "end point" ascertained. When the combustion of the adulterant was over, the crucible was cooled and weighed; the loss in weight represents the volatile matter and the adulterant. The crucible was again heated to above 900°C. in a muffle furnace, with free circulation of air, till the graphite was burnt off completely, and the ash content determined. The decrease in weight is due to graphite.

The experimental results with crystalline graphite from Ceylon are shown in Table I and with amorphous graphite from Mysore in Table II.

It will be noted from Tables I and II that the error did not exceed 2 per cent, which may be tolerated in commercial analysis, particularly when no alternative method is available.

The author's thanks are due to Principal R. P. Sinha and Prof. S. K. Bose for their interest in the work, and to Prof. H. N. Das Gupta for criticism and helpful suggestions.

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# Letters to the Editor

## A LICHEN (*PARMELIA TINCTORUM*) ON A JAVA MONUMENT

THE ADVERSE EFFECT OF THE GROWTH OF lichens has been previously recorded. A recent report<sup>1</sup> by us related to the damage done to sandal-wood trees near Waltair by *Ramalina tayloriana*. In that connection it was suggested that besides physical injury due to the penetration of the hold-fasts of this lichen, the action of the toxic components (lichen acids) secreted by the lichen should also be considered to play an important part. This general idea seems to have a wider applicability than originally expected. A very similar case is discussed below.

Recently, Mr. K. R. Srinivasan, Superintendent, *Archaeological Survey of India*, gave us small samples of cryptogamic vegetation that he collected from the walls of Barabudur, the world famous Buddhist monument of central Java which he visited last year. The most prominent of these are lichens which are reported to occur over large areas not only defacing the sculptured panels, but also obscuring the finer details. They seem to affect the surface fabric by their continued growth and activity. Further, during the process of cleaning the sculptures mechanically, the dried lichens remove along with them the adhering substratum, thereby disfiguring many of them. One of these samples was large enough (3 gm.) for detailed study. It has been identified as *Parmelia tinctorum* Despr. and is identical botanically with an Indian sample which has been studied earlier<sup>2</sup> and shown to be exceptional in that it contained besides atranorin (0.8 per cent) and lecanoric acid (5.0 per cent) some amount of norstictic acid (1.0 per cent) also. An extraordinary feature of the Barabudur sample is that it contains a very high percentage (20.3 per cent) of lecanoric acid besides a small amount of atranorin. Such a high percentage was recorded earlier by Hesse<sup>3</sup> also in another case of this lichen under the name *Parmelia coralloides*. This is of special importance in the present context because lecanoric acid is sufficiently strong and water soluble to be reckoned with in affecting soft and particularly calcareous

structures. When there is such a high percentage of it, the chances of its being secreted by the lichen and causing damage to the monument are considerable.

### Experimental

The air-dried powdered lichen (3.0 gm.) was extracted repeatedly with ether (250 c.c.) in the cold and the extract distilled in order to remove the solvent. The dried extract was digested with hot petroleum ether (100 c.c.) and filtered. The filtrate was concentrated to a very small bulk and allowed to stand overnight when colourless prismatic crystals (*c.* 20 mg.) melting at 193°-195°C. separated. In alcoholic solution, it gave a brown-red colour with ferric chloride and dissolved in aqueous potassium hydroxide producing a yellow colour. Mixed melting point with an authentic sample of atranorin was undepressed. The petroleum-ether insoluble solid was dissolved in cold acetone and to the clear solution was added chloroform in excess when a colourless crystalline solid (610 mg.) melting at 170°-175°C. was obtained. This was crystallized from a mixture of acetone and chloroform (1:3), when it came out as colourless needles melting at 175°-176°C. It was easily soluble in sodium bicarbonate solution. In alcoholic solution, it gave a violet colour with a drop of ferric chloride and with bleaching powder a bluish, red colour. It was identified as lecanoric acid by mixed melting point determination with an authentic sample of the compound obtained earlier from the Indian lichen sample.

The lichen powder left after ether extraction was again extracted with hot acetone and the concentrated extract allowed to stand for a few days. No crystalline solid separated. The solvent was completely removed and the small quantity of residue left was qualitatively tested. It gave no reaction with alcoholic ferric chloride, bleaching powder and potassium hydroxide solution indicating that no other component of the depsidone type was present in any detectable amount.

T. R. SESHADRI

S. SANKARA SUBRAMANIAN

*Andhra & Delhi Universities*

July 15, 1949

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### SOME OCCURRENCES OF KYANITE IN THE AJMER-MERWARA DISTRICT, RAJPUTANA

THE AUTHOR HAS EXAMINED SEVERAL OCCURRENCES of kyanite near Ajmer and a brief account of the geology and mode of occurrence of these deposits is given below. It may be noted that these occurrences lie in the extreme north-west corner of map-sheet No. 45 J/15 and the southern portion of the sheet lying to the north of it.

The geology of the area consists of the following formations and rock types :

1. Serpentinized peridotites
2. The schistose complex
3. Pegmatites and quartz veins
4. White marble
5. Quartzites
6. Alluvium

*Serpentinized Peridotites* — Small hills of serpentinized peridotites occur north of the Bhudol-Naulakha road *en route* to Gudos ( $26^{\circ} 29' : 74^{\circ} 46'$ ). A little asbestos is associated with these rocks and the occurrence has been described already<sup>1</sup>.

*Schistose Complex* — The author has given the name of "schistose complex" to a series of schists, the most dominant being the mica schists, but it must be observed that there has been considerable metamorphism of these rocks by the intrusion of pegmatites and quartz veins. As a result biotite-talcose-schist, biotite-tremolite-schist, biotite-actinolite-schist, garnetiferous-schist, staurolite-schist, kyanite-schist, etc., have developed. These schists, in places, are highly foliated and well cleaved, the cleavage angle varying from  $50^{\circ}$  to  $80^{\circ}$  and dipping towards E.N.E. and N.E. In places, these schists are highly contorted.

From an economic point of view, these schists with injections of pegmatites and quartz veins are important as emeralds, kyanite and talc are associated with them.

*Pegmatites and quartz veins* are seen intrusive into the schists. The pegmatites comprise the following types : (i) quartz-tourmaline-pegmatites ; (ii) quartz-tourmaline-biotite-pegmatites ; and (iii) mica (muscovite) pegmatites.

Their strike was observed to vary from N.E.-S.W. to N.N.E.-S.S.W.

Veins of hard quartz-tourmaline-schist also occur associated along with those of pegmatites. Its mineral composition is similar to that of the pegmatites ; it is only schistose in character. It is a modification of quartz-tourmaline-pegmatite with which biotite is associated.

*Marble* — About one furlong S.W. of Thakur Ram Nath Singh's bungalow near the village of Akhri ( $26^{\circ} 33' : 74^{\circ} 48'$ ) coarsely crystallized white marble occurs. The country being flat, the rock crops out in places and the quarries have been opened to extract marble. It is quite an extensive outcrop and extends almost beyond the Gegal-Khodan road. The rock is dressed into blocks of various sizes, the biggest being about 3' square. It is coarsely jointed and thus it can be easily taken out into large blocks.

*Limestone* — About 150 yards S.S.E. of the well between Muhami ( $26^{\circ} 30' 30'' : 74^{\circ} 46' 44''$ ) and Bubani villages, a band of limestone occurs which extends for some distance and has a N.N.E.-S.S.W. strike. Nearby, crystals of staurolite were collected from the surface. The schists forming the country rock are highly contorted in places.

*Quartzites* — Quartzites form conspicuous hills and a series of ridges in the area. Being of no economic value, at least so far as the occurrence of kyanite is concerned, they have not been described here.

*Occurrences of Kyanite* — A little south-west of the peridotite hill near the Bhudol-Naulakha road, mica schists are exposed which have a N.N.E.-S.S.W. strike. In these schists a pit had been sunk about 50 yards from the road and about a quarter mile south-west of Gudos village ( $26^{\circ} 29' : 74^{\circ} 46'$ ) which yielded kyanite which, in places, had bright bluish colour. Close by, a considerable quantity of vein quartz was observed. Specimens of talcose and greenish chloritic schists were also observed. The schists are highly foliated in places.

Nearby, a few other pits had been sunk in which kyanite was found occurring as lentils in the biotite schists. Some garnetiferous schist also occurs.

Kyanite occurs again in pockets in the mica schists a little further north-east on the western slopes of the peridotite hill.

About half a mile, almost due north of the village of Bhudol ( $26^{\circ} 29' 30'' : 74^{\circ} 45' 18''$ ), a quartz vein with a north-south strike is

intrusive into the schists. Kyanite occurs here associated with this quartz vein in the form of veins and pockets. Small inclusions of kyanite occur in quartz which, as a result of jointing, breaks into large and small pieces. A trench, about 25' in length, in a N.N.W.-S.S.E. direction and about 4.5' in width had been dug. The thickness of the quartz vein was observed to vary from 2' to 4' and the kyanite is developed at the contact for about 2.5' from the quartz vein with inclusions of biotite.

The country rock is a biotite-schist and the following section is observed in the trench. On the top there is a layer of overburden, about 9" in thickness, in which small irregular pieces of quartz, etc., are found embedded in red earth. This is underlain by a layer of decomposed schists with whitish calcareous concretions, having a thickness of 2'. These are succeeded by biotite schists. Kyanite is enclosed in the quartz vein and is also developed at the contact. A few maunds of kyanite had been recovered when the working was stopped.

Close by other quartz veins occur with which some tourmaline-quartz rock is also associated.

It would appear from the above that the biotite schist mainly represents the country rock into which quartz veins are intrusive and kyanite has developed at the contact. It, therefore, definitely appears that as a result of this igneous activity alumina has

been supplied by the biotite schists, while the quartz veins have furnished silica and the two at the contact have developed kyanite. It may be noted that in one specimen of kyanite a small inclusion of sapphire was observed. This evidently represented a small excess of alumina which had crystallized as such. It may be noted that in this neighbourhood many quartz veins occur and their contacts certainly deserve further attention.

It has been noted by A. M. Heron<sup>2</sup>: "There was an output of 3 tons of kyanite from Ajmer-Merwara district in 1931, but the locality is not known." In all probability the kyanite must have been recovered from one of these localities.

It may be noted that the area is easily accessible. It is about 9 miles in a direct line from Ajmer and can be approached from Ajmer-Kishengarh road whence a cart track, which is fit for motor traffic also, will take one to these occurrences.

H. L. CHHIBBER

*Banaras Hindu University*  
May 28, 1949

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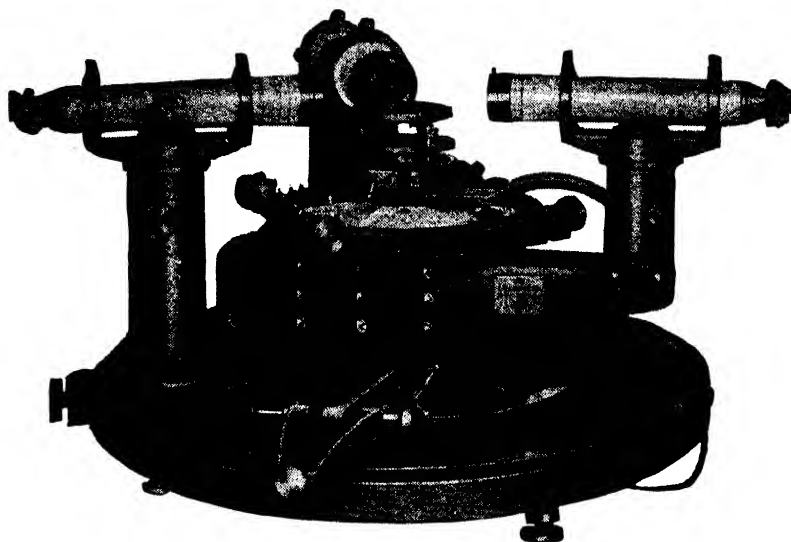
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# Chemical Examination of *Picrorhiza kurrooa* Benth.—Part I

RAM PRAKASH RASTOGI, VISHWA NATH SHARMA & SALIMUZZAMAN SIDDIQUI

*Chemical Laboratories, C.S.I.R., Delhi*

**P**ICRORHIZA *kurrooa* Benth. (*Scrophulariaceae*; Hindi: *kutki* or *katki*) grows from Kashmir to Sikkim at an altitude of 5,000 to 10,000 feet. The rhizome and the roots are extremely bitter to taste and have been recommended as a substitute for the imported gentian roots. They are extensively used in the indigenous system of medicine as a tonic, a febrifuge and an anti-periodic. In spite of the considerable importance attached to the plant, its chemical investigation has been limited to a preliminary examination by Dymock and Hooper<sup>1</sup> who reported the presence of a "brown, resinoid glucoside", picrorhizin (yield, 15 per cent), which, on hydrolysis with 1 per cent hydrochloric acid, yielded a reducing sugar, and an aglucone, picrorhizetin, a reddish-brown, brittle, resinous, tasteless mass. They further obtained the free aglucone, picrorhizetin, from the plant along with a wax melting at 61°C. Chopra and Ghosh<sup>2</sup> also noted the presence of the bitter glucoside in the drug but were unable to isolate any crystalline product.

As a result of the present investigations, the following products have been isolated from the roots in a crystalline form:

1. A glucosidal bitter principle, kutkin,  $C_{23}H_{28}O_{12}(C_{23}H_{24}O_{10} \cdot 2H_2O)$ ; m.p. 211°C.; yield 3.4 per cent on the weight of the dry roots; bitter in a dilution of 1 in 7,500.
2. A non-bitter product, kurrin,  $C_9H_{18}O_9$ ; m.p. 166°C.; yield, 0.5 per cent on the weight of the dry roots.
3. Vanillic acid,  $C_8H_8(OH)(OCH_3)(COOH)$ ; yield, 0.1 per cent on the weight of the dry roots.

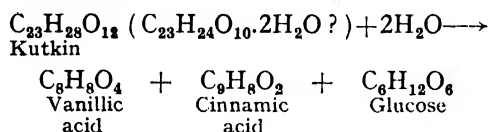
4. Kutkiol,  $C_{40}H_{82}O$ ; m.p. 118°C.; occurring as the acetate,  $C_{40}H_{81}OOC.CH_3$ , m.p. 74°C.; yield, 0.06 per cent on the weight of the dry roots.
5. Kutki-sterol,  $C_{24}H_{40}O$ ; m.p. 124°C.; yield, 0.18 per cent on the weight of the dry roots;  $[\alpha]_D^{25} = -39.0^\circ$  in 1 per cent chloroform solution.

As described in detail in the experimental, the method employed for the isolation of kutkin and kurrin was based on the difference in their solubilities in alcohol. The isolation of kurrin was comparatively easier, but kutkin could be obtained in a pure crystalline form after a lengthy process of purification employing mixed solvents.

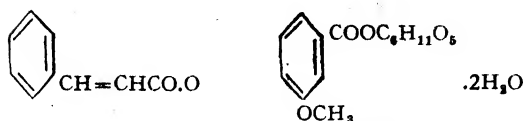
*Constitution of Kutkin,  $C_{23}H_{28}O_{12}$* —Kutkin is soluble in water, methanol or alcohol in the hot, but insoluble in other organic solvents. It contains one methoxy group, but no phenolic group, as it does not give any colouration with ferric chloride in alcoholic solution. It dissolves in dilute alkali or ammonia with a yellow colouration but is not precipitated out on acidification. In conformity with its glucosidal character, it reduces Fehling solution after hydrolysis with dilute mineral acids. Kutkin is, moreover, a  $\beta$ -glucoside, as it is also hydrolysed by emulsin under the usual conditions. No crystalline aglucone could be obtained from the products of its hydrolysis with mineral acids due to considerable resinification occurring during hydrolysis. On treatment, however, with dilute aqueous alkali at the ordinary temperature or with saturated baryta solution on the water bath, it yielded 2 crystalline substances melting at 202°C. and 133°C. respectively, along with glucose



which was identified through its osazone. The yields of the two crystalline products were very much better when the hydrolysis was carried out with baryta solution. The substance melting at 202°C. could also be obtained in a small yield through hydrolysis with 1 per cent acetic acid in a sealed tube at 125°C. The analytical data and the chemical characteristics of the substances melting at 202°C. and 133°C. as well as of their derivatives showed them to be vanillic and cinnamic acids respectively. This finding was confirmed through the mixed melting point determinations of the two products and their derivatives with authentic samples of vanillic and cinnamic acids and their corresponding derivatives. The course of hydrolysis may be represented by the following equation :



Accordingly, kutkin as a glucose ester of cinnamoyl-vanillic acid should correspond to the molecular formula  $\text{C}_{23}\text{H}_{24}\text{O}_{10}$  (requires C, 60.0; H, 5.2). The C and H values for the substance dried at 100°C. *in vacuo* over phosphorus pentoxide (C, 55.0; H, 5.3) are in agreement with the molecular formula  $\text{C}_{23}\text{H}_{28}\text{O}_{12}$  (requires C, 55.6; H, 5.6). This could, however, be accounted for by the presence of 2 molecules of water of crystallization in kutkin, which are not removable at 100°C. Such an explanation should find support from a similar behaviour in the case of Gentiopikrin<sup>3</sup>, a glucoside from *Gentiana lutea* L., which splits off its 1 molecule of water of crystallization only on hydrolysis and not under the usual conditions of drying. Further studies to elucidate this point are in progress but the following tentative structure of kutkin may be put forward at this stage :



As it was of interest to find out whether vanillic and cinnamic acids occur in the drug in a free state, a sample of the powdered drug was Soxhleted with ether after defatting it through extraction with petrol ether. The

etheral extract, on working up in the usual manner, yielded vanillic acid (yield 0.1 per cent), but no cinnamic acid could be obtained from it.

*Kurrin*,  $\text{C}_9\text{H}_{18}\text{O}_9$  — Kurrin is readily soluble in water, sparingly so in methanol and insoluble in the other organic solvents. It does not give a reaction for a free carboxylic group, contains one methoxy group, and 5 (or 6) active hydrogens as determined after Zerewitinoff. It is optically inactive. Kurrin does not reduce Fehling solution either before or after hydrolysis with dilute sulphuric acid. On benzylation, both after Schotten-Baumann and with benzoyl chloride in pyridine solution, it yields only a penta-benzoyl derivative. The analytical data of the acetyl derivative, however, obtained with acetic anhydride and fused sodium acetate indicate the formation of a tetra acetyl derivative with the intramolecular loss of two molecules of water. Action of phenyl hydrazine on kurrin also results in the formation of a triphenyl hydrazone with the intramolecular elimination of two molecules of water. It could not be methylated with either methyl iodide and silver oxide or with dimethyl sulphate and alkali. With formaldehyde and formic acid, no methylation could be effected, but a crystalline product was obtained by this reaction, which conforms to the molecular formula  $\text{C}_9\text{H}_{14}\text{O}_6$  and does not contain the methoxy group present in the original substance. It is recovered unchanged on fusion with 50 per cent potash. Kurrin yields a crystalline semi-carbazone but its micro-N-values are too high for any conceivable formula. Further investigations to clarify these points and elucidate the chemical constitution of kurrin are in progress.

*Fatty Portion* : *Kutkiol*,  $\text{C}_{40}\text{H}_{81}\text{OH}$  — The petrol ether extract of the drug yielded a fatty portion which, on keeping in benzoyl solution, gave kutkiol acetate,  $\text{C}_{42}\text{H}_{84}\text{O}_2$ , m.p. 74°C. On hydrolysis with alcoholic potash, this product gave acetic acid and an aliphatic alcohol,  $\text{C}_{40}\text{H}_{81}\text{OH}$ , melting at 118°C., which has been provisionally named as kutkiol.

On saponification of the fatty residue left on removal of the solvent from the mother liquors of kutkiol acetate, a sterol,  $\text{C}_{24}\text{H}_{40}\text{O}$ , was obtained from the unsaponifiable portion, which melts at 124°C. and appears to be different from the known sterols. It has been provisionally named as kutki-sterol.

## Experimental

**Isolation** — 1 kg. of the finely powdered roots was extracted with petroleum ether and the defatted material was repeatedly percolated with alcohol till the roots were no longer bitter in taste. The total alcoholic percolates were concentrated *in vacuo* below 50°C., and the dark-brown, viscid concentrate was freed of the residual fat by digestion with petroleum ether. The combined petroleum ether extracts, on removal of the solvent, gave 9.6 gm. of fatty matter.

The petroleum ether insoluble residue was taken up in alcohol and the alcoholic solution was kept in the cold, when it gave a crystallize of crude kurrin which was filtered and washed with alcohol. It melted at 158°C. (decomp.), yield 0.5 per cent.

The combined filtrate and washings from kurrin were treated with ether and petroleum ether which precipitated out darkish non-bitter matter. The supernatant reddish-brown solution was freed of the solvent first on the water bath at the ordinary pressure and finally *in vacuo*. The residue was taken up in alcohol and subjected to a similar procedure of purification through ether and petroleum ether. The alcohol-ether-petroleum ether solution obtained at this stage was kept in the cold, when a crystalline deposit of crude kutkin (m.p. 196°C. decomp.) slowly formed, which was filtered and washed with a mixture of alcohol and ether. The combined filtrate and washings were evaporated off and the residue was again dissolved in alcohol and treated with ether-petroleum ether, when a second crop of the crude kutkin was obtained (total yield 3.4 per cent). The final mother liquors were bitter in taste but failed to give any further crystalline product either from any of the organic solvents or on repetition of the process of purification through alcohol, ether and petroleum ether.

**Kutkin**,  $C_{23}H_{28}O_{12}$  — Crude kutkin (m.p. 196°C. decomp.) was repeatedly crystallized from alcohol and methanol when it was finally obtained in the form of colourless clusters of needles melting at 211°C. Kutkin is soluble in water, methanol and alcohol in the hot, and insoluble in acetone, ether and ethyl acetate. In 1 per cent alcoholic solution, it showed  $[\alpha]_D^{25} = -165.0^\circ$ . Kutkin readily reduces Fehling solution

after hydrolysis with 0.1 per cent sulphuric acid. It does not give any effervescence with sodium bicarbonate solution nor any colouration with ferric chloride. It dissolves in dilute alkali and ammonia with a yellow colouration which turns pink on keeping overnight, but the substance cannot be precipitated out on acidification of the alkaline or the ammoniacal solution. It dissolves in concentrated sulphuric acid with a deep violet colouration and in concentrated nitric acid with a light-yellow colour. It did not suffer any loss in weight on drying at 100°C. *in vacuo* over phosphorus pentoxide and the dried sample, on analysis, gave C, 55.0; H, 5.3;  $OCH_3$  (after Zeisel), 2.0.  $C_{23}H_{28}O_{12}$  requires C, 55.6; H, 5.6;  $OCH_3$  (for one), 3.0 per cent.

**Acetyl Kutkin**,  $C_{31}H_{36}O_{16}$  — 0.2 gm. of kutkin, 5 c.c. of acetic anhydride and a few drops of pyridine were refluxed at 135°C. for 2 hr. After working up the reaction product in the usual manner, the acetyl derivative was crystallized from dilute methanol (yield 0.26 gm.). It crystallizes in clusters of needles melting at 170°C. and soluble in methanol, alcohol, acetone, ether and benzene, insoluble in water, and petroleum ether. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 54.9; H, 5.2; M.W. (Rast), 621. Tetra-acetyl kutkin,  $C_{31}H_{36}O_{16}$ , requires: C, 56.0; H, 5.4; M.W., 664. Penta-acetyl kutkin,  $C_{33}H_{38}O_{17}$ , requires C, 56.0; H, 5.3, M.W., 706.

### Hydrolysis of Kutkin (Isolation of Vanillic & Cinnamic Acids from the Hydrolysate —

**Hydrolysis with Baryta Solution** — A solution of 0.5 gm. of kutkin in 15 c.c. of saturated baryta solution was heated on the water bath for half an hour. The resulting pink solution was saturated with carbon dioxide and the precipitated barium carbonate filtered off. The clear filtrate was acidified with dilute sulphuric acid and extracted with ethyl acetate. The ethyl acetate solution was washed with water, dried over anhydrous sodium sulphate and freed of the solvent. The residue (0.24 gm.), on crystallization from chloroform, yielded two crystalline products, one (0.15 gm.) sparingly soluble in chloroform in the cold and melting at 200°C., and the other (0.07 gm.) fairly soluble in chloroform in the cold and melting at 126°C.

The aqueous acidic layer from the above working was treated with excess of barium

carbonate, filtered and tested for sugars, as described later.

The crystalline product melting at 200°C. was repeatedly crystallized from chloroform when it finally yielded colourless needles melting at 202°C., soluble in methanol, alcohol, acetone, ether and ethyl acetate, and insoluble in water, petrol ether and benzene. It did not give any colouration with ferric chloride, but dissolved in sodium carbonate solution with effervescence. It is soluble in dilute alkali with a yellow colouration, and is precipitated out unchanged on acidification of the solution. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 56.8; H, 4.7; M.W. (Rast), 151;  $\text{OCH}_3$  (after Zeisel), 8.0;  $\text{C}_8\text{H}_8\text{O}_4$  requires C, 57.1; H, 4.8; M.W., 168;  $\text{OCH}_3$  (for one), 8.9 per cent. On admixture with an authentic sample of vanillic acid, it did not show any depression in the melting point.

The acetyl derivative (m.p. 141°C.) of the product was prepared in the usual manner with acetic anhydride and fused sodium acetate. On analysis, it gave C, 56.9; H, 5.2; M.W. (Rast), 218;  $\text{C}_{10}\text{H}_{10}\text{O}_5$  requires C, 57.1; H, 4.8; M.W., 210. On admixture with acetyl vanillic acid, it did not give any depression in the melting point.

Demethylation of the product melting at 202°C. with hydriodic acid gave a phenolic acid (m.p. 195°C. decomp.), identical with protocathechuic acid.

The second crystalline product melting at 126°C., obtained from the hydrolysate, after a few crystallizations from hot water finally melted at 133°C. It was soluble in water and most of the organic solvents. It did not give any colouration with ferric chloride, and dissolved in bicarbonate solution with effervescence. It absorbed bromine in the cold in chloroform solution. After drying to constant weight at 62°C. *in vacuo* over phosphorus pentoxide, it gave C, 72.7; H, 5.6; M.W. (Rast), 154;  $\text{C}_9\text{H}_8\text{O}_2$  requires C, 72.9; H, 5.4; M.W. 148.

On oxidation with potassium permanganate in alkaline solution, it gave a smell of benzaldehyde, and from the reaction mixture a crystalline acid was obtained which was identified as benzoic acid. On admixture with an authentic sample of cinnamic acid, the crystalline product melting at 133°C. did not show any depression in the melting point.

*The Sugar Component*—The aqueous solution, obtained after extraction of vanillic and cinnamic acids and neutralization with barium carbonate in the manner described earlier, yielded a residue on evaporation, which gave an osazone of golden-yellow needles melting at 203°C. The osazone did not show any depression in the melting point on admixture with glucosazone.

For a quantitative estimation of the sugar, kutkin was hydrolysed with 5 per cent aqueous sodium hydroxide by keeping for 48 hr. at the room temperature. The pink-coloured reaction mixture was heated with Fehling solution and the precipitated cuprous oxide was filtered in a Gooch crucible, washed, dried and weighed. The weight of the cuprous oxide corresponded to 31.7 per cent of glucose.  $\text{C}_{23}\text{H}_{28}\text{O}_{12}$  requires (for one molecule of glucose) 36.2 per cent.

A 2 per cent aqueous solution of kutkin, on keeping with emulsin at 40°C. for 30 min., turned yellow and reduced Fehling's solution.

*Action of Dilute Acids & Alkali on Kutkin*—Hydrolysis of kutkin with 5 per cent aqueous sodium hydroxide on keeping for 48 hr. at room temperature also yielded the two crystalline products obtained through hydrolysis with baryta solution, but the total yield of the two products was only 30 per cent on the weight of kutkin as against 63 per cent of the theoretical and 48 per cent by the baryta hydrolysis.

Hydrolysis of kutkin with 0.2 per cent of sulphuric acid on the water bath led to considerable resinification and blackening of the solution, and no crystalline product could be obtained from the reaction mixture.

Dilute acetic acid (1 to 3 per cent), in a sealed tube at 125°C., hydrolysed kutkin, and from the products of hydrolysis vanillic acid could be isolated in a yield of 20 per cent. No cinnamic acid could, however, be obtained from this hydrolysate.

*Isolation of Free Vanillic Acid from the Roots*—The ether extract of a sample of the defatted drug was concentrated on the water bath and shaken with dilute sodium bicarbonate solution. The bicarbonate solution was acidified, extracted with ether, the ethereal layer was washed with water and dried over anhydrous sodium sulphate, and freed of the solvent. The light brown residue gave, through chloroform, colourless

needles of vanillic acid melting at 202°C. (yield 0.1 per cent on the weight of the dried powdered roots).

**Kurrin**,  $C_9H_{18}O_9$ .—Crude kurrin, melting at 158°C. (decomp.), was repeatedly crystallized from hot dilute methanol, when it finally yielded the pure product in the form of colourless, silky needles melting at 166°C. Kurrin is soluble in cold water, sparingly soluble in hot methanol and nearly insoluble in other organic solvents. It does not reduce Fehling solution either before or after hydrolysis with dilute sulphuric acid and does not give any effervescence with sodium carbonate solution. It readily dissolves in concentrated sulphuric acid and concentrated nitric acid giving colourless solutions. It is optically inactive. Attempts to methylate kurrin with dimethyl sulphate and alkali or with methyl iodide and silver oxide after the method of Irvine and Purdie were not successful. Kurrin was recovered unchanged on heating with dilute sulphuric acid or fusion with 50 per cent potassium hydroxide at 190°C. After drying to constant weight *in vacuo* over phosphorus pentoxide, it gave C, 39.7; H, 7.3;  $OCH_3$  (after Zeisel), 5.5; active H (Zerewitinoff), 1.7, 2.1; mean, 1.9.  $C_9H_{18}O_9$  requires C, 40.0; H, 6.6;  $OCH_3$  (for 1), 5.5; active H, (for 5) 1.8; (for 6 H), 2.2 per cent.

**Penta-benzoyl-kurrin**,  $C_{44}H_{38}O_{14}$ .—0.1 gm. of Kurrin was dissolved in 10 per cent sodium hydroxide and 1 c.c. of benzoyl chloride was slowly added to the solution with vigorous shaking and cooling. After completion of the reaction, the resulting product was extracted with ether and the ethereal solution was washed with dilute alkali and then water, dried over anhydrous sodium sulphate and freed of the solvent on the water bath. The residue, on crystallization from methanol, yielded the benzoyl derivative in the form of colourless clusters of needles, melting at 142°C.; yield 0.29 gm. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 66.6; H, 4.5;  $C_{44}H_{38}O_{14}$  requires C, 66.7; H, 4.8 per cent.

0.1 gm. of the substance on benzoilation in dry pyridine solution with 2 c.c. of benzoyl chloride also gave the benzoyl derivative obtained above in a yield of 0.26 gm.

**Acetylation of Kurrin (Anhydro-tetra-acetyl Kurrin)**.—A mixture of 0.1 gm.

of substance, 1 gm. of fused sodium acetate and 2 c.c. of acetic anhydride was heated on a water bath for 2 hr. The reaction mixture was diluted with water and the acetyl derivative, obtained as a whitish powder, was filtered, washed with water and crystallized from alcohol. It melts at 120°C. (yield 0.12 gm.), is soluble in methanol, alcohol, benzene, ether and ethyl acetate, and insoluble in water, chloroform and petrol ether. After drying to constant weight at 61°C. *in vacuo* over phosphorus pentoxide, it gave C, 49.6; H, 6.2; M.W. (cryoscopic in benzol), 398.  $C_{17}H_{22}O_{11}$  (tetra-acetyl kurrin with intramolecular elimination of two molecules of water) requires C, 50.7; H, 5.5; M.W., 402.

0.1 gm. of the substance, on acetylation with 2 c.c. of acetyl chloride in dry pyridine solution, also gave an identical acetyl derivative (yield 0.11 gm.).

**Action of Phenyl Hydrazine on Kurrin**.—An aqueous solution of 0.1 gm. of the substance was heated on the water bath for 2 hr. with 1 gm. of phenyl hydrazine hydrochloride and 1 gm. of sodium acetate. The reaction mixture was acidified with dilute acetic acid and extracted with ether. The ethereal layer was washed with water, dried over anhydrous sodium sulphate and freed of the solvent. The residue, on crystallization from ligroin, yielded clusters of yellowish-orange needles melting at 128°C.; yield 0.07 gm. After drying to constant weight at 61°C. *in vacuo* over phosphorus pentoxide, it gave C, 64.5; H, 6.5; N, 17.0 per cent.  $C_{27}H_{32}O_6N_6$  (triphenyl hydrazone of kurrin with intramolecular elimination of two molecules of water) requires C, 64.2; H, 6.3; N, 16.6 per cent.

#### *The Fatty Portion*—

**Kutkiol Acetate**.—The petroleum ether extract of the powdered roots, after removal of the solvent under vacuo, yielded a greenish-brown oil (9.6 gm.) with a characteristic smell which, on keeping in benzol solution, gave a crystalline deposit of the crude kutkiol acetate. After a few crystallizations from hot ethyl acetate, it was finally obtained as clusters of needles melting at 74°C. (yield 0.06 per cent). Kutkiol acetate is soluble in petroleum ether, benzene, ether and ethyl acetate in the hot, and insoluble in methanol and alcohol. After drying to constant weight *in vacuo* over phosphorus pentoxide, it gave C, 81.4; H, 13.7; M.W. (Rast), 535.  $C_{42}H_{54}O_2$  requires C, 81.3; H, 13.5; M.W., 620.

On hydrolysis with alcoholic potassium hydroxide, it yielded an alcohol, kutkiol, melting at 118°C. and acetic acid. Kutkiol, on analysis after drying to constant weight *in vacuo* over phosphorus pentoxide, gave C, 82.6; H, 14.7; M.W. (Rast), 542.  $C_{40}H_{82}O$  requires C, 83.0; H, 14.1; M.W., 578.

*Kutki-sterol* — 10 gm. of the residue left on removal of the solvent from the mother liquors of kutkiol acetate was saponified with 15 per cent alcoholic potash by heating on the water bath for 3 hr. The alkaline solution was diluted with water and repeatedly extracted with ether and the ethereal layer was washed free of alkali, dried over anhydrous sodium sulphate and freed of the solvent. The orange-coloured residue (3.9 gm.), on repeated crystallizations from alcohol, gave colourless needles of the sterol melting at 124°C.

The sterol gave the characteristic Liebermann-Burachard colour reactions for a phytosterol. It is soluble in petroleum ether, ether, ethyl acetate and chloroform, sparingly soluble in alcohol, and insoluble in methanol and water. In 1 per cent chloroform solution, it gave  $[\alpha]_D^{25} = -39.0^\circ$ . It gave a digitonide melting at 245°C. (decomp.). After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, the sterol gave C, 83.6; H, 11.8; M.W. (Rast), 384.  $C_{24}H_{40}O$  requires C, 83.7; H, 11.6; M.W., 344.

The acetyl derivative prepared by heating the sterol with acetic anhydride and fused sodium acetate on the water bath melted at 110°C.

The percentage of sterol in the fat was determined by the digitonide method of Windaus. 25 c.c. of 1 per cent solution of digitonin in 95 per cent alcohol were added to the unsaponifiable matter obtained from 5 c.c. of the fat, dissolved in the same solvent. After standing overnight the precipitate was filtered on a weighed Gooch crucible, washed with alcohol, dried at 110°C. and weighed (0.88 gm.; equivalent to 0.18 per cent on the weight of the dry roots).

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## Chemical Composition of the Flowers of *Butea superba*

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THE flowers, seeds and root bark of *Butea frondosa* have been used in Indian medicine and have already been examined chemically. The closely related plant, *Butea superba*, has also been used in medicine; the flowers and bark have been claimed to be useful in scorpion stings and snake bites. The roots, which are considered in Siam to have rejuvenating

properties, were examined and the presence of two glucosides noted<sup>1</sup>. Preliminary physiological experiments indicated the presence of an oestrogenic substance and also a poisonous substance<sup>2</sup>. In the present note, the results of the examination of the flowers of *Butea superba* obtained from the forests of the Godavari Agency are presented. They were studied on the same lines as were

adopted for *Butea frondosa* flowers<sup>3,4</sup>. From the alcoholic extract, on concentration, was obtained a heavy precipitate of a glycoside which was insoluble in ether, chloroform and acetone. When this was purified and crystallized from aqueous alcohol, it was colourless, melted at 193°-194°C. (decomp.), and agreed in all its properties with butrin. The identity was established by hydrolysis with dilute acid whereby a mixture of butin and butein were obtained. Confirmation was provided by methylation using dimethyl sulphate and anhydrous potassium carbonate in dry acetone medium and final hydrolysis of the methylated product yielding 2:4-dimethyl-butein.

In certain experiments it was found possible to isolate mono-methyl butrin which was identical with a similar product obtained by the action of diazomethane on butrin<sup>5</sup>. This, on hydrolysis, yielded a mixture of 4'-methyl-butin and 4'-methyl butein. This type of partial methylation yielding a methyl ether of a flavanone without a free hydroxyl group in the 5-position is ordinarily unusual<sup>6</sup> but in the present case, probably owing to the sparing solubility of butrin and its mono-methyl-ether in acetone, its formation and isolation are possible. Earlier, Lal<sup>7</sup> reported the formation of mono-methyl butrin by the action of methyl iodide and potassium carbonate in absolute methyl alcoholic medium. A typical methylation experiment yielding this result is described here. Finely powdered butrin (1.0 gm.), anhydrous potassium carbonate (3.0 gm.), dimethyl sulphate (1 c.c.) and pure dry acetone (40 c.c.) were refluxed together for 30 hr. The acetone was removed and the residue was dissolved in 25 c.c. of water and set aside. A colourless, crystalline mass slowly separated out. This was filtered and crystallized from hot water. The substance melted at 222°C., did not give any colour with ferric chloride, but gave a bright-violet colour with magnesium powder and hydrochloric acid. It was found to be identical with mono-methyl butrin obtained by methylating butrin with diazomethane.

The yield of butrin from *B. superba* flowers is almost the same as from *B. frondosa* flowers. From the mother liquor left after the separation of butrin could be isolated butein and butin. Thus there is close resemblance in every respect between the two flowers. The *superba* flowers have a more marked orange-red colour but

this seems to be due to the presence of carotenoids.

With reference to the medicinal properties of these flowers, it may be appropriate to mention here the recent finding<sup>8</sup> that butein and similar chalcones have marked bacteriostatic properties which are also possessed by related flavanones. Consequently, we have now examined the toxic properties of butein and butin on fish. It is found that in a concentration of 100 mg. per litre (a dilution of 1 in 10,000), butein exhibits toxic effects (fish turning upside down) in the course of 2 hr. and 50 per cent of the fish employed do not recover but die even after their removal to fresh water. On the other hand, butin is considerably less toxic since, even at a concentration of 200 mg. per litre, it exhibits no toxic effect on fish during 24 hr. Thus, it would appear that hydroxy-flavanones, in virtue of their lower toxicity, may serve as better anti-bacterials. A number of related chalcones, flavanones and their methyl ethers were examined recently<sup>9</sup> with respect to their toxic properties and the results reported. With chalcones, the toxic symptoms set in more slowly but they are more persistent; the methoxy-chalcones are less toxic than the isomeric flavanones whereas the reverse is the case when a number of hydroxyl groups are present. As soon as all the hydroxyl groups of the chalcones are methylated, the toxicity increases considerably. The present study of butin and butein agree in general with these conclusions.

### Summary

The flowers of *Butea superba* yield the same crystalline components as those of *Butea frondosa*, the predominant product being butrin. Butin is far less toxic to fish than butein.

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# Some Polymerized Products from Castor Oil

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WHILE carrying out investigations on the dehydration of castor oil with various catalysts, it was found that selenium did not catalyse dehydration of the oil to any appreciable extent. An interesting phenomenon, however, was observed when the treated oil was allowed to cool. The oil, on cooling, was found to have thickened and become almost solid like wax.

Action of catalysts on polymerization of oils has been widely studied but these studies mainly refer to the production of drying oils. Sulphur and sulphur-containing compounds have been employed by Gardner<sup>1</sup> in the bodying of tung oil to prevent gelation. Harfer<sup>2</sup> used selenium for the same purpose. Parkin<sup>3</sup> has patented the use of diaryl and dialkyl disulphides. The action of liquid sulphur monochloride on oils like castor oil and rape-seed oil has been studied by Kaufman, Baltes and Mardner<sup>4</sup>. Recently, drying oil polymers have been developed having the properties of natural rubber such as Norepol, Agripol<sup>5</sup>, Vulprene<sup>6</sup>, etc., from soya-bean or linseed oil.

The thickened product obtained by us is different from the products referred to above and does not appear to have been studied so far. No reference in literature could be traced in respect of the use of selenium as a polymerization catalyst.

A study of the polymerized products formed under varying experimental conditions

has been, therefore, made and the results are given in this communication.

## Experimental

The oil was thoroughly mixed with 1 per cent metal powder and heated at different temperatures for varying periods. The products obtained were examined for their softening points, acid, iodine, saponification and acetyl values, and solubility in ethyl alcohol. The results are shown in Table I.

Increase in the acid value indicates increased splitting and decomposition of the glycerides with increase in temperature. The iodine value is stationary, indicating that there is no change in the unsaturation of the glycerides. Fall in the acetyl value indicates some reduction in the hydroxyl groups. Solubility in alcohol at all stages shows that the hydroxyl groups are not appreciably affected.

The above experiments show that: (1) products of varying consistencies are obtained; (2) heating at 200°C. appears to produce maximum hardening; and (3) the products have rather high acidity, a feature which is undesirable for most of the uses to which these products are likely to be put.

Experiments were then carried out to obtain products of low acidity by heating the oil in an atmosphere of carbon dioxide for different intervals of time and testing the resultant products.

TABLE I—CATALYST 1 PER CENT

No.*	TEMPERATURE OF REACTION °C.	TIME OF HEATING hr.	NATURE OF PRODUCT		ACID VALUE	IODINE VALUE (Wijs')	SAPONIFICATION VALUE	ACETYL VALUE	SOLUBILITY IN 95% ETHYL ALCOHOL (1:1)	SOFTENING POINT °C.
			on cooling	after 24 hr.						
1.	150-160	1	Liquid, greenish	Solid, greenish	16.4	85.7	182.4	138.7	Completely soluble	45
2.	150-160	2	do	do	17.6	84.8	183.9	138.1	do	54
3.	150-160	3	do	do	19.2	84.9	184.9	137.2	do	52
4.	200-210	1	do	Solid, light green	21.2	84.4	183.4	134.3	do	55
5.	200-210	2	do	do	22.1	84.3	184.6	132.1	do	66
6.	200-210	3	do	do	22.9	84.2	185.7	130.2	do	64
7.	250-260	1	Thick liquid brownish	Solid, slightly reddish brown	31.0	84.6	185.2	129.5	do	50
8.	250-260	2	do	do	34.5	84.3	186.6	126.0	do	43

\* In experiments 1, 2, and 3, the catalyst was collected at the bottom in powder form at the end of the reaction, while in experiments 4 to 8, it was in a molten state and formed a lump. On cooling, the mass was found to be porous and could be easily powdered.

TABLE II

Temperature 200°C.; carbon dioxide atmosphere;  
catalyst selenium (a) 1%; (b) 2%

No.	INTERVALS AT WHICH SAMPLES WERE DRAWN, min.	CONSIS- TENCY	ACID VALUE	SOFTENING POINT (CAPILLARY TUBE METHOD) °C.	REMARKS
	Untreated oil	Liquid	2.3		
1.	(a) 30	Solid after keeping overnight	2.2	40.6	Suitable for use as petroleum jelly and vaseline
	(b) 30	do	2.0	43.3	
2.	(a) 60	do	2.2	49.51	
	(b) 60	do	2.1	50	
3.	(a) 90	do	2.4	54.5-57	Suitable for use as wax substitute and lubrica- ting grease
	(b) 90	do	2.2	55.6	
4.	(a) 120	do	2.5	68.70	
	(b) 120	do	2.2	60	
5.	(a) 150	do	2.7	60.62	
	(b) 150	do	2.4	57.2	
6.	(a) 180	do	2.8	57.60	
	(b) 180	do	2.5	53.9	

The results of these experiments are given in Table II.

From the above results, the following points emerge:

1. In an atmosphere of carbon dioxide, the acidity of the final product is low.

2. In conformity with results of the previous findings, after a certain period of heating, the melting point of the product begins to fall.

3. The maximum hardening is reached after about 2 hr.; the product obtained has a somewhat higher softening point; and

4. there is no advantage in using higher percentages of the catalyst.

Larger samples of hardened castor oil of the consistency of petroleum jelly or vaseline were prepared using 1 per cent selenium catalyst in a carbon dioxide atmosphere. The product was faintly greenish yellow and had an acid value of 2.9.

Samples of the product were sent to the Ordnance Laboratories, Kanpur, for being tested whether it conforms to the Indian Stores Department specification for petroleum jelly, and to the Principal, Medical College, Agra, for being tested as a substitute for vaseline. The report from the Ordnance Laboratories was that the product conformed in all respects to the Indian Stores Department Specification G/O-101/86 for mineral jelly except for the lower softening point and higher organic acidity.

The report of the Principal, Medical College, Agra, shows that the first sample sent to him was found to cause irritation when applied to the eyes and had an acid

smell. The product was refined to remove the acidity by caustic soda treatment and subsequent washing and drying. The moisture-free neutral material was then treated with 3 per cent bleaching earth and 1 per cent of "Decol" for half an hour at 80°C., and filtered hot. The filtered oil was then treated for an hour with superheated steam (at 250°C.) under vacuum and at a temperature of 100° to 125°C. The improvement resulting from the treatment is shown in Table III.

The refined sample was found satisfactory as a base for medicinal ointments. It did not promote bacterial growth, caused no irritation and possessed no objectionable odour.

As selenium is rather costly, it was considered advisable to study whether the catalyst can be used continuously with a view to lengthen its useful life and obtain a maximum yield of the polymerized product. To ascertain whether the reaction could be carried out continuously, fresh quantities of the raw oil were run into the flasks containing the catalyst mass left over from previous trials. Samples were drawn out, as before, at intervals of 1 hr. The results obtained are given in Table IV.

The results show that the action of the catalyst used for a second time was not quite satisfactory. It was mainly due to the lack of thorough intermixing of the catalyst and the oil. Accordingly, the residual catalyst was thoroughly mixed with the oil by grinding before the heating operation.

TABLE III

	PRODUCT BEFORE TREATMENT	PRODUCT AFTER TREATMENT
Colour	Faint green	No change
Acid value	2.9	0.8
Smell	Acrid	Odourless

TABLE IV

Temperature, 200°C.; castor oil, 500 gm.; catalyst, 5 gm. selenium;  
carbon dioxide atmosphere

No.	INTERVAL AT WHICH SAMPLES WERE DRAWN hr.	CATALYST LEFT OVER FROM 1ST SERIES OF EXPERIMENTS USED	CATALYST LEFT OVER FROM 2ND SERIES OF EXPERIMENTS USED
1.	1	Product: liquid after 24 hr. cooling	Product: liquid after 24 hr. cooling
2.	2	do	Product: viscous li- quid after 24 hr. cooling
3.	3	Product: viscous liquid after 24 hr. cooling	Product: very thin paste after 24 hr. cooling
4.	4	do	Product: thin paste after 24 hr. cooling



### Experimental

Castor oil (5 kg.) was saponified by slight warming with 1 kg. of caustic soda dissolved in 2 litres of water. The resulting pale-white soap contained 20 per cent moisture and about 1 per cent excess alkali. 200 gm. lots of this soap were mixed thoroughly with weighed amounts of different basic materials, transferred to round-bottom flasks with separating funnels and distilling heads, and heated in an air bath. The temperature was raised gradually. Much hydrogen gas was evolved in the beginning and care was taken not to lose the octyl alcohol which distilled over along with some water. The water was separated from the alcohol and poured back into the flask through the separating funnel at the rate of 2 or 3 drops a minute. The temperature was gradually raised to 200°C. till no more octyl alcohol distilled over. This process takes 50-70 hr. continuous heating. The crude octyl alcohol thus collected was fractionally distilled and the fraction between 175°-180°C. was pure secondary octyl alcohol. The solid product which remained in the flask was suspended in water, acidified with dilute hydrochloric acid, boiled and filtered hot. The filtrate, on cooling, deposited crystals of sebacic acid (m.p. 133°-134°C.). They were filtered and dried at 40°-50°C. The percentage of octyl alcohol and sebacic acid obtained are given in Table I.

TABLE II

PERIOD OF HEATING hr.	YIELD OF SEBACIC ACID %
4	25.5
8	53.0
12	53.8

The results show that a maximum yield of sebacic acid is obtained when castor oil soap is heated in the presence of 20-30 per cent of caustic soda. The yield of secondary octyl alcohol, however, is maximum when castor oil soap is heated with 30 per cent sodium carbonate. Besides a higher yield, the alcohol is purer.

In order to find out the effect of heating under pressure, 200 gm. of the soap were mixed with 60 gm. of caustic soda and the mixture heated in an autoclave at 260°C. at a steam pressure of 700 lb./sq. in. for different lengths of time. The yields of sebacic acid are given in Table II.

Heating for 8 hr. under pressure will give a higher yield of sebacic acid than pyrolysis under ordinary pressure.

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## Extraction of Potash from Felspar

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**F**ELSPAR has been located in Hyderabad State in abundant quantity in Raichur, Mahboobnagar, Gulberga and Nalgonda districts<sup>1</sup>. Potash, a valuable fertilizer, is locked up in felspar. No commercial process is as yet available for its recovery from this mineral. The dry processes for the extraction of potash from felspar require high temperature and special equipment. The wet processes

consisting of digestion of felspar with a calcium compound under pressure are comparatively simpler. Besides, in the latter case, the residual material after the recovery of potash is a valuable by-product which can be used in the preparation of cement and bricks.

Ross<sup>2</sup> obtained a 90 per cent yield of potash when a mixture of felspar (5 gm.) and lime (8 gm.) is digested in a nickel-

steel bomb of 250 c.c. capacity, under a pressure of 10-15 atm. The purpose of this investigation is to study the above process using commercial lime and larger quantities of the materials and also to study the effect of using basic calcium nitrate in the place of lime.

### Experimental

The Golconda felspar contains 11.3 per cent  $K_2O$  and 5.0 per cent  $Na_2O$ , while Mahboobnagar variety consists of 9.86 per cent  $K_2O$  and 4.67 per cent  $Na_2O$ . The Golconda felspar which is richer in potash content was used in all the experiments.

The felspar was heated in an oven to about  $700^{\circ}$ - $800^{\circ}C.$ , quenched in cold water, crushed to pass through 120 mesh and analysed. The results of the analysis was as follows :

	%
Silica	64.58
Alumina	17.71
Iron oxide	0.50
Calcium oxide	0.80
Magnesium oxide	—
$K_2O$	11.30
$Na_2O$	5.00

Felspar (120 mesh powder), commercial quicklime (120 mesh powder) and enough water were added into an autoclave and thoroughly mixed. The steel autoclave

(4 litres capacity) was enclosed in an oil bath, and provided with a safety valve, a pressure gauge and a thermometer and heated with a stove. The digestions were carried out at 100, 150, 200 and 250 lb. per sq. in. pressure. After digestion, the resulting slime was filtered through a Buchner funnel, the residue was washed 4 times with hot water and the potash in the filtrate estimated by the cobalt-nitrite method<sup>3</sup>. The results of experiments with different samples of commercial lime, containing 54.6, 70 and 90 per cent of  $CaO$ , at different pressures and intervals of time have been given in Table I.

It will be seen from the results that the yield of potash increases with increase in pressure, 90.8 per cent of the potash contained in felspar being recovered when a mixture of 225 gm. of lime (containing 200 gm. of calcium oxide) and 100 gm. of felspar powder is digested at 250 lb. per sq. in. pressure for 6 hr. (expt. 10). Increase in the quantity of the felspar in the digestion mixture results in a lower yield under similar conditions (cf. expts. 6, 7 and 8). The results of experiments 10, 11 and 12 show that the yield of potash is proportional to the calcium oxide present in the mixture. Calcium carbonate present in commercial samples of lime does not act on felspar. The yield is enhanced when calcium carbonate

TABLE I

EXPT. NO.	WT. OF FELSPAR gm.	WT. OF QUICKLIME gm.	PURITY OF LIME %	CALCIUM PRESENT AS $CaO$ gm.	REACTION PRESSURE lb./sq. in.	TIME hr.	YIELD OF POTASH %
1	100	200	54.6	78	100	4	10.4
2	100	200	54.6	78	100	6	23.4
3	50	143	70	71	150	4	61.0
4	50	143	70	71	150	6	66.0
5	50	143	70	71	200	6	78.0
6	50	143	70	71	250	6	88.0
7	100	286	70	142	250	6	71.8
8	200	570	70	284	250	6	63.3
9	200	570	70	284	250	10	70.0
10	100	225	90	144	250	6	90.8
11	100	112	90	72	250	6	46.3
12	100	56	90	36	250	6	23.3

TABLE II

EXPT. NO.	WT. OF FELSPAR gm.	WT. OF QUICKLIME gm.	PURITY OF LIME	NITRIC ACID (66%) C.C.	TOTAL CALCIUM IN THE MIXTURE gm.	REACTION PRESSURE lb./sq. in.	TIME hr.	YIELD OF POTASH
								Calculated
13	100	185	54.6	120	104	150	45.0	
14	100	185	54.6	120	104	150	55.5	
15	100	185	54.6	120	104	150	62.8	
16	100	92	54.6	60	52	150	21.35	22.0 (13)
17	100	92	54.6	60	52	150	28.7	27.8 (14)
18	100	92	54.6	60	52	150	30.4	31.4 (15)
19	50	70	70	60	46	150	30.0	30.52 (3)
20	50	70	70	60	46	200	53.6	50.54 (5)
21	50	70	70	60	46	250	58.3	57.02 (6)
22	100	107	90	120	73	250	49.3	46.01 (10)

is converted into a reactive calcium compound such as calcium nitrate.

Enough nitric acid was added to the mixture of felspar, lime and water so as to form a mixture ultimately consisting of felspar, calcium nitrate and calcium oxide. The quantities of felspar, lime, nitric acid employed, the conditions of digestion and yield of potash are given in Table II.

When felspar is digested with a mixture of calcium oxide and calcium nitrate, the yield of potash is proportional to the total calcium content. The observed results show good agreement with the calculated values. Further work is in progress to find out the part played by calcium oxide and calcium nitrate in the reaction.

#### Utilization of Sludge

Bricks made from the sludge remaining over after the extraction of potash, under a pressure of 2 tons per sq. in., and steamed for 8 hr. at a pressure of 100 lb./sq. in. possess a crushing strength of 2,200 lb. per sq. in.

Superior quality sand-lime bricks can be made from the sludge when mixed with suitable amount of sand and water. Bricks were prepared with 55 gm. of sludge, 365 gm. of sand and 25 c.c. of water and steamed as described above. The crushing strengths of the bricks compare well with those of first-grade sand-lime bricks (TABLE III).

#### Summary

1. The yield of potash is 90.8 per cent when felspar and lime (90 per cent CaO)

TABLE III

PURITY OF LIME USED FOR DIGESTION %	CRUSHING STRENGTH lb./sq. in.	CRUSHING STRENGTH; A.S.T.M. STANDARDS FOR SUPERIOR GRADE (SW) S.L. BRICK lb./sq. in.
90	5130	3500-4500
70	3820	

mixture containing felspar : calcium oxide as 1 : 2 by weight is digested at 250 lb./sq. in. pressure.

2. The yield of potash is proportional to calcium content of lime. Calcium carbonate present in the commercial samples of lime is inert. The yield is improved by converting calcium carbonate to calcium nitrate by the addition of nitric acid. The yield is then proportional to the total calcium content.

3. The sludge obtained after recovery of potash can be used in the preparation of sand-lime bricks.

#### Acknowledgement

I wish to express my sincere thanks to Dr. M. Qureshi for suggesting the problem and for his guidance. My thanks are also due to Dr. D. S. Datar for constant help and supervision.

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## Letters to the Editor

### ANALYTICAL USES OF BENZOHYDROXAMIC ACID

THE ANALYTICAL APPLICATIONS OF BENZOHYDROXAMIC ACID in qualitative and quantitative inorganic analysis are described in this note. The reagent gives deep-coloured, stable, water-soluble complexes with a number of cations. It promises to be of

great use in the detection of traces of metals like uranium, vanadium and molybdenum.

The potassium salt of the reagent is easily obtained by reacting a methyl alcohol solution of ethyl benzoate with hydroxylamine in the presence of potassium hydroxide. The reagent is neutralized with dilute hydrochloric acid before use. Best results are obtained in nearly neutral media as the

development and intensity of colour are adversely affected by low or high pH.

The sensitivity and the limit of concentration for uranium and vanadium with this reagent are superior to the limits obtained with the other standard "spot" test reagents in use (potassium ferrocyanide and hydrogen peroxide respectively). The results obtained with molybdenum also compare favourably with those obtained with phenylhydrazine reagent.

Tests carried out on a "spot" plate with 3 per cent neutral aqueous solution of the reagent gave the following results:

TABLE I

CATION	REAGENT	SENSITIVITY	LIMIT OF CONCENTRATION
Uranium	Benzohydroxamic acid	0.5 $\mu$	1 : 200,000
	Pot. ferrocyanide	0.9 $\mu$	1 : 50,000
Vanadium	Benzohydroxamic acid	0.2 $\mu$	1 : 250,000
	Hydrogen peroxide	2.5 $\mu$	1 : 20,000
	Ferric chloride Dimethylglyoxime	1.0 $\mu$	1 : 50,000
Molybdenum	Benzohydroxamic acid	1.0 $\mu$	1 : 200,000
	Phenylhydrazine reagent	0.1 $\mu$	1 : 300,000

**Uranium**—A deep-orange colour is obtained in weak acid medium, while in weak ammoniacal solution, an orange precipitate soluble in excess of ammonia is produced.

**Vanadium**—A deep-purple colour is given by pentavalent vanadium in neutral or slightly alkaline solutions.

**Molybdenum**—A golden-yellow colour is produced in weakly ammoniacal medium.

The test for vanadium is of especial importance as it promises to be a convenient method for colorimetric detection and estimation of vanadium in magnetite ores, specially because the deep colour of the iron complex can be easily masked with hydrofluoric acid.

The conditions for the quantitative determination of the cations are being investigated.

The work was undertaken at the suggestion of Dr. J. Gupta, Asst. Director, National Chemical Laboratory, to whom our thanks are due.

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*Delhi*  
July 11, 1949

## A NOTE ON THE CHEMICAL EXAMINATION OF NIM (*MELIA AZADIRACHTA*) EXUDATE

THE CHEMICAL EXAMINATION OF NIM EXUDATION was undertaken as a part of the scheme of systematic investigation on nim (*Melia azadirachta*; *Melia indica*)<sup>1-6</sup>. The exudation is a rare, seasonal occurrence in comparatively older trees, and is reputed in the indigenous systems of medicine as an alterative tonic. The sap was collected locally in the months of December-January. It had a strong smell of fermented liquor with the characteristic odour of nim, and was opalescent in character. It was slightly sweetish in taste and acidic to litmus.

The freshly collected exudate had a specific gravity, 1.0525 at 19°C. and a pH, 5.0. It contained total solids, 12.5 per cent; reducing sugar (calculated as glucose), 0.5 per cent; total acids (calculated as acetic acid), 0.8 per cent. The sap was dextro-rotatory but exact determination of the optical rotation was not possible because of the opalescent character of the liquid which could not be got over by repeated filtration. Analysis of the ash from the total solids, precipitated by the addition of alcohol, showed the presence of phosphate, sulphate, iron and calcium. An essential oil, giving a qualitative test for sulphur, was isolated from the steam distillate of the exudation, in a comparatively poor yield. Acetic acid was identified in the sap distillate.

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August 22, 1949

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### A NOTE ON THE CHEMICAL EXAMINATION OF NIM (*MELIA AZADIRACHTA*) TRUNK-BARK

CHEMICAL EXAMINATION OF THE NIM TRUNK-bark, reputed as a febrifuge in the indigenous systems of medicine, has been carried out in continuation of the systematic investigation on the various parts of the nim tree (*Melia azadirachta*; *Melia indica*). The crystalline bitter constituents, nimbin and nimbinin, and the amorphous bitter, nimbidin, isolated earlier from the nim oil<sup>1</sup> and the root-bark<sup>2</sup>, have also been obtained from the alcoholic extract of the trunk-bark in an yield of 0.04, 0.001 and 0.4 per cent respectively. The oil-soluble bitter, nimbidol (loc. cit.), was not, however, found to be present in the trunk-bark. An essential oil having similar characteristics to that isolated from the nim blossoms<sup>3</sup> has also been obtained from the petrol-ether soluble fraction of the alcoholic extract in an yield of 0.02 per cent. The unsaponifiable fraction of the fatty portion of the extract yielded nimbosterol (loc. cit.) in an yield of 0.03 per cent. The tannin content of the bark has been found

to be ca. 6 per cent on the weight of the dry bark.

The isolation of an amorphous glucoside from the nim trunk-bark has been reported earlier by Sen<sup>4</sup>, but following the procedure adopted by him for the isolation of the substance, it has only been possible to obtain a product consisting of tannin material contaminated with the bitter principles of the nimbidin series.

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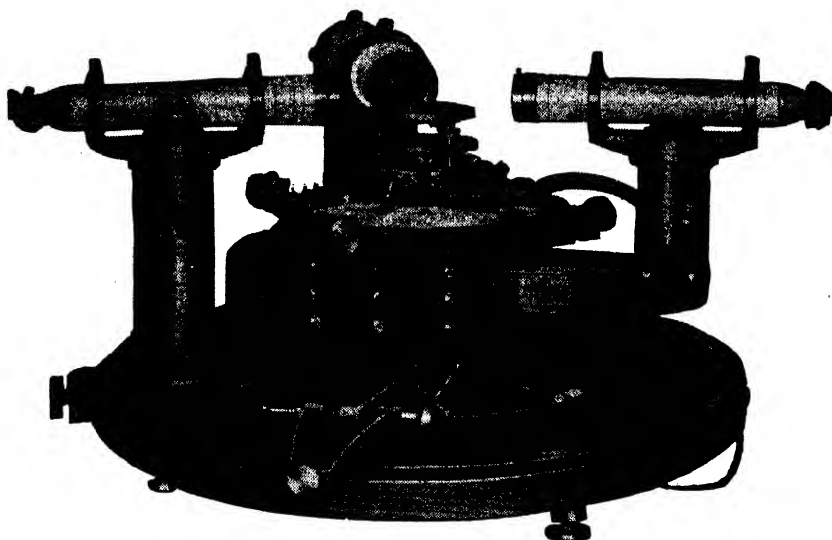
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# Studies in the 3-phenyl Coumarin Series: Part I—Synthesis of Some New Members of the Series

PRITHVI RAJ BHANDARI, JOGENDRA LAL BOSE & SALIMUZZAMAN SIDDIQUI

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WHILE 2- and 3-phenyl- $\gamma$ -benzopyrones, namely flavones and isoflavones on the one hand, and simple coumarins and furo-coumarins on the other, form important groups of natural products, the phenyl- $\alpha$ -benzo-pyrones, namely 3- and 4-phenyl coumarins have not so far been found to occur in nature. This is particularly striking in reference to 3-phenyl coumarins and isoflavones, as in both cases the 3-aryl radical is attached to the  $\alpha$ -carbon atom with respect to the carbonyl group of the pyrone ring, and an interchange of the position of  $-\text{CH}=\text{}$  and  $-\text{CO}-$  with the corresponding adjustment of the double bond would convert an isoflavone to a 3-phenyl coumarin, or the latter into the former. This is illustrated by the interesting work of Baker<sup>1</sup> relating to the attempted conversion of the isoflavan derivative, anhydro-catechin tetramethyl ether, to a tetramethoxy-isoflavone by oxidation, which actually resulted in the formation of a tetramethoxy-3-phenyl coumarin. A further illustration of the same point was given by the attempted synthesis of 7-hydroxy isoflavone through the condensation of  $\alpha$ -formyl-phenyl-acetonitrile with resorcinol resulting in the formation of 7-hydroxy 3-phenyl coumarin<sup>2</sup>.

The work embodied in the present paper was started in the first instance to exclude the possible 3-phenyl coumarin structure for biochanin A, one of the main constituents of Bengal gram (*Cicer arietinum* Linn.) germ<sup>3</sup>, the constitution of which was later established as 4'-methoxy 5, 7-dioxy isoflavone on the basis of degradation experiments<sup>4</sup>,

and confirmed by its synthesis by Shriner and Hull<sup>5</sup>, in connection with their work on the constitution of prunetin. Studies in the synthesis of 3-phenyl coumarins were subsequently extended further, in view of the lack of any systematic work in the field and also in consideration of the fact that some members of the series have been found to possess high haemostatic activity.

Since the first synthesis of the parent compound of the series, namely 3-phenyl coumarin by Oglialoro<sup>6</sup>, no work was done in the field for a long time until Bargellini and co-workers<sup>7</sup> synthesized a number of 3-phenyl coumarins in connection with their work on the constitution of some  $\alpha$ -benzopyrones, synthesized by Jacobson and Ghosh<sup>8</sup> and presumed by them to be  $\gamma$ -benzo-pyrones. Later Bargellini, by the synthesis of 4', 5, 7-trioxy and trimethoxy 3-phenyl coumarins, which proved to be different from prunetol, the demethylated product of prunetin and methyl prunetin respectively, excluded the possible 3-phenyl coumarin constitution for prunetin, the isoflavone constitution for which was subsequently established by a synthesis of prunetol (genistein) by Baker and Robinson<sup>9</sup>.

Meerwein and co-workers<sup>10</sup> have also recently synthesized a few 3-phenyl coumarins in the course of their studies on the reaction of aromatic diazo compounds with  $\alpha$ - $\beta$ -unsaturated carbonyl compounds.

Out of the three general reactions available for the synthesis of 3-phenyl coumarins, namely (1) Perkin's reaction; (2) Knoevenagel's reaction; and (3) the reaction of aromatic diazo compounds with coumarin, the



first was found in the present studies to be most suitable for the synthesis of oxy-3-phenyl coumarins. In this method the oxy-coumarins are initially obtained as acetoxy derivatives, and the method of deacetylation had, therefore, to be carefully chosen for ensuring good yields. In this connection deacetylation with concentrated sulphuric acid at 0°C. was found to be better than either deacetylation with dilute sulphuric acid at higher temperatures or with alkali.

All the hydroxy and methoxy-3-phenyl coumarins synthesized in the course of the present work were found to exhibit strong fluorescence even in high dilutions in various solvents as well as in alkali and concentrated sulphuric acid. The fluorescence data of the coumarins along with their melting points are given in Table I.

### Experimental

**4'-Acetoxy-3-phenyl coumarin**—A mixture of salicylaldehyde (0.3 gm.), sodium salt of *p*-hydroxyphenylacetic acid (0.5 gm.) and acetic anhydride (6 c.c.) was refluxed in an oil bath for 25 hr. at 150°-160°C. After cooling to the room temperature, the reaction mixture was poured in an excess of cold water and left overnight. The crude product (0.35 gm.) was collected and repeatedly crystallized from dilute alcohol (charcoal) when it was finally obtained in colourless prismatic rods, m.p. 183°C. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate, chloroform and benzene and very sparingly soluble in petroleum ether.

Found: C, 73.2; H 4.6; required for  $C_{17}H_{14}O_4$ : C, 72.9; H, 4.3 per cent.

**4'-Oxy-3-phenyl coumarin**—4'-Acetoxy-3-phenyl coumarin (0.2 gm.) was taken in a small flask and cooled in ice. Cold

concentrated sulphuric acid (0.5 c.c.) was added to it, drop by drop, mixing the acid with the solid coumarin thoroughly after the addition of each drop when a dark, homogeneous, viscous mass was obtained. After keeping for 10 min., crushed ice was added to the reaction mixture and the separated coumarin was filtered and thoroughly washed with water. The crude product (0.16 gm.) was crystallized a number of times from alcohol (charcoal) when finally the pure coumarin was obtained in colourless prismatic rods, m.p., 202°C. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate and chloroform, sparingly soluble in benzene and insoluble in petroleum ether.

Found: C, 75.7; H, 4.2; required for  $C_{15}H_{10}O_3$ : C, 75.6; H, 4.2 per cent.

On methylation with methyl iodide in acetone solution in presence of freshly desiccated potassium carbonate, it yields *p*-methoxy-3-phenyl coumarin, m.p., 143°-144°C. There was no depression in the melting point on admixture with a sample of 4'-methoxy-3-phenyl coumarin prepared from salicylaldehyde and sodium homoanisate.

**4',7-Diacetoxy-3-phenyl coumarin**—A mixture of resorcydaldehyde (0.5 gm.), sodium salt of *p*-hydroxy phenyl acetic acid (0.72 gm.) and acetic anhydride (10 c.c.) was refluxed for 30 hr. at 170°-180°C. On working up in the usual manner, 0.5 gm. of the crude product was obtained. It was crystallized a number of times from acetone-methanol mixture and was finally obtained in small colourless needles, m.p., 213°-214°C. It is soluble in methanol, alcohol, acetone, ether, ethyl acetate, chloroform and benzene, and insoluble in petroleum ether.

Found: C, 67.2; H, 4.2; required for  $C_{19}H_{14}O_6$ : C, 67.4; H, 4.1 per cent.

TABLE I—FLUORESCENCE CHARACTERISTICS OF THE VARIOUS COUMARINS

No.	COUMARIN	M.P. °C.	ACETONE	ALCOHOL	ETHER	CONCENTRATED SULPHURIC ACID	5% POTAS- SIUM HYDROXIDE (AQUEOUS)
1.	4'-acetoxy-3-phenyl coumarin	183	Nil	Nil	Nil	Yellow	Yellow
2.	4'-oxy-3-phenyl coumarin	202	Blue with violet tinge	Bluish violet	Bluish violet	Pale yellow	Yellow
3.	4', 7-diacetoxy-3-phenyl coumarin	213-214	Very faint bluish violet	Faint violet	Very faint violet	Yellow	Yellow
4.	4', 7-dioxy-3-phenyl coumarin	320-321	Bluish violet	Greenish yellow Violet	Violet	Yellow	Greenish yellow Yellow
5.	4', 7-dimethoxy-3-phenyl coumarin	182	Violet	Violet	Violet	Yellow	Yellow
6.	4'-methoxy-7-acetoxy-3-phenyl coumarin	178	Bluish violet	Bluish violet	Bluish violet	Yellowish green	Greenish yellow
7.	4'-methoxy-7-oxy-3-phenyl coumarin	232	Bluish violet	Bluish violet	Bluish violet	Yellow	Greenish yellow
8.	4'-methoxy-5, 7-diacetoxy-3- phenyl coumarin	182	Bluish violet	Blue	Blue	Pale yellow	Yellow
9.	4'-methoxy-5, 7-dioxy-3-phenyl coumarin	280-282	Bluish violet	Blue	Bluish violet	Deep yellow	Yellow

**4',7-Dioxy-3-phenyl coumarin** — 4',7-Diacetoxy-3-phenyl coumarin (0.2 gm.) was deacetylated with concentrated sulphuric acid (0.5 c.c.) at 0°C. On working up in the usual manner, 0.15 gm. of the crude product was obtained. It was crystallized from alcohol (charcoal) and then a number of times from methanol-acetone mixture and was finally obtained in aggregates of colourless needles, m.p., 320°-321°C. (with decomposition). It is soluble in methanol, alcohol, acetone, ether and ethyl acetate and insoluble in chloroform, benzene and petroleum ether.

Found: C, 70.5; H, 4.1; required for  $C_{15}H_{10}O_4$ : C, 70.9; H, 3.9 per cent.

**4',7-Dimethoxy-3-phenyl coumarin** — 4',7-dioxy-3-phenyl coumarin (0.1 gm.) was dissolved in dry acetone (10 c.c.) and freshly desiccated potassium carbonate (0.2 gm.) was added to it followed by methyl iodide (0.5 c.c.). The mixture was refluxed at 60°C. for 4 hr. and was left overnight at the room temperature. The solvent was removed *in vacuo*, and the residue was washed with water and crystallized from alcohol (charcoal) when the methoxy coumarin was obtained in colourless needles, m.p., 182°C.; yield, 0.06 gm. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate, chloroform and benzene and very sparingly soluble in petroleum ether.

Found: C, 72.1; H, 4.7;  $OCH_3$ , 21.3; required for  $C_{17}H_{14}O_4$ : C, 72.3; H, 4.9;  $OCH_3$ , 21.9 per cent.

**4'-Methoxy-7-acetoxy-3-phenyl coumarin** — A mixture of resorcyaldehyde (0.35 gm.), sodium homoanisate (0.5 gm.) and acetic anhydride (5 c.c.) was refluxed for 20 hr. at 160°-170°C. in an oil bath and the reaction product was poured into water and left overnight. The crude crystalline product was collected and repeatedly crystallized from acetone when the pure coumarin was finally obtained in aggregates of colourless needles, m.p. 178°C., yield, 0.3 gm. It is soluble in alcohol, methanol, acetone, ether, ethyl acetate, chloroform and benzene and insoluble in petroleum ether.

Found: C, 69.6; H, 4.7; required for  $C_{18}H_{14}O_5$ : C, 69.7; H, 4.5 per cent.

**4'-Methoxy-7-oxy-3-phenyl coumarin** — 4'-Methoxy-7-acetoxy-3-phenyl coumarin (0.2 gm.) was deacetylated with concentrated sulphuric acid (0.5 c.c.) at 0°C. After keeping at the room temperature (24°C.) for 5 min., crushed ice was added to the solution when the deacetylated coumarin separated out. On repeated crystallizations

from methanol, it was finally obtained in colourless needles, m.p., 232°C.; yield, 0.15 gm. It is soluble in alcohol, methanol, acetone, ether and ethyl acetate, sparingly soluble in chloroform and insoluble in benzene and petroleum ether.

Found: C, 71.5; H, 4.5;  $OCH_3$ , 11.0; required for  $C_{16}H_{12}O_4$ : C, 71.6; H, 4.5;  $OCH_3$ , 11.6 per cent.

On methylation of the coumarin with methyl iodide in the usual manner, a product identical with 4',7-dimethoxy-3-phenyl coumarin, m.p., 182°C., was obtained.

**4'-Methoxy-5,7-diacetoxy-3-phenyl coumarin** — A mixture of phloroglucinol aldehyde (0.5 gm.), sodium homoanisate (0.75 gm.) and acetic anhydride (10 c.c.) was refluxed at 160°-170°C. for 30 hr. After cooling, water was added to the reaction mixture and left overnight. The solid which separated out was crystallized from alcohol (charcoal) and then from slightly diluted acetone when the coumarin was finally obtained in colourless silky needles, m.p., 182°C.; yield, 0.43 gm. It is soluble in alcohol, methanol, acetone, ethyl acetate, benzene, ether and petroleum ether and sparingly soluble in chloroform.

Found: C, 64.9; H, 4.4; required for  $C_{20}H_{16}O_7$ : C, 65.2; H, 4.3 per cent.

**4'-Methoxy-5,7-dioxy-3-phenyl coumarin** — 4'-methoxy-5,7-diacetoxy-3-phenyl coumarin (0.1 gm.) was deacetylated with concentrated sulphuric acid at 0°C. The deacetylated coumarin crystallized from methanol-acetone mixture in small pale-yellow needles, m.p., 280°-282°C., yield, 0.07 gm. It is soluble in alcohol, methanol, acetone, ether and ethyl acetate, sparingly soluble in chloroform and benzene and insoluble in petroleum ether.

Found: C, 67.2; H, 4.1;  $OCH_3$ , 10.6; required for  $C_{16}H_{12}O_5$ : C, 67.6; H, 4.2;  $OCH_3$ , 10.9 per cent.

On methylation of 4'-methoxy-5,7-dioxy 3-phenyl coumarin with methyl iodide 4', 5, 7 trimethoxy 3-phenyl coumarin crystallizing from alcohol in small colourless needles was obtained. It melted at 162°-163°C., as against 163°-165°C., recorded in the literature.

The phenolic aldehydes required for the synthesis were prepared by Karrer's method<sup>11</sup>, namely by the action of cyanogen bromide on phenols, and homoanisic acid and *p*-hydroxy phenylacetic acid by hydrolysing the corresponding derivatives of phenylacetone nitriles.

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## Ultrasonic Absorption in Unassociated Liquids

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THE absorption in the case of polyatomic gases can be attributed to the excitation of vibrational levels of the molecules by ultrasonic waves, to the lag between the excited molecules returning to their normal levels and to the time period of vibration of ultrasonic waves, known as the relaxational phenomenon. In 1938 Kneser<sup>1</sup> explained the phenomenon of absorption in the case of liquids by assuming the same mechanism ; that is, he assumed that in the case of liquids also the vibrational levels are excited. The vibrational contribution at any particular temperature is calculated from spectroscopic data, viz. the frequencies of the most intense Raman lines and infra-red bands. It is an observed fact that the intensity of Raman lines decreases when the temperature is raised<sup>2</sup>. It follows, therefore, that with the rise of temperature the number of excited molecules increases and these molecules, when exposed to ultrasonic waves, will not be in a favourable condition to take up energy from the incident waves, i.e. the absorption coefficient will decrease. This leads to a negative temperature coefficient of absorption which is not verified by experiment.

The authors are of the opinion that the mechanism of absorption in the case of liquids is not entirely due to vibration of atoms, but that the structural changes in the quasi-crystalline state of liquids play an important part in the absorption of ultrasonic waves, and suggest that the phenomenon of absorption and viscosity

are related and that both can be described in terms of "hole theory" of liquid state formulated by Furth<sup>3</sup> and Frankel<sup>4</sup> and developed by Auluck and Kothari<sup>5</sup>. Briefly, the conception of the liquid state may be described in the following manner.

The simplest model suggested by Furth is that the liquids also, like crystals, have short-distance order arrangement and geometrical regularity. In this lattice arrangement there are certain sites which are unoccupied by molecules. These unoccupied places are filled by molecules from outside. Similarly, in the lattice arrangement a molecule jumps out and causes a vacancy. At a given temperature a statistical equilibrium is maintained. These unoccupied places or sites have been given the name "holes". Increase of temperature results in the destruction of holes and the geometrical structure and at the boiling point the structure of the holes is completely deranged. Hence it follows that the quasi-crystalline liquid structure must be endowed with a certain amount of energy when the holes are present. The energy can be calculated if the geometrical structure is known, otherwise we have to calculate it from other data. For instance, the energy at any temperature can be calculated from the rate of evaporation :

$$\dot{v} = S \sqrt{\frac{kT}{2\pi m}} \frac{A}{RT}$$

where  $a$  is the number of molecules escaping from the surface of the body per unit time and under unit surface into the

empty space,  $S$  the number of molecules per unit volume,  $m$  the mass of the molecules,  $k$  the Boltzman constant,  $T$  the absolute temperature and  $A$  the work function required to remove  $N$  molecules from the interior of the body to infinite distance.

In the case of ultrasonic waves, it is suggested that the statistical equilibrium at a particular temperature is disturbed and the number of holes increase. The required energy to increase the number of holes is taken from the incident sound waves ( $\Delta T \sim 11^\circ\text{C}$ . for a sound intensity  $10 \text{ W/cm}^2$  at  $1 \text{ Mc}$ ).

If during the cycle the sound wave completes one period, the holes return to their equilibrium value and no absorption will result. If, on the other hand, there is a lag, then the energy taken away by the holes will not be given back to the sound waves and this will amount to loss of energy from the sound waves.

Having in mind the structure of a liquid described above, we have to calculate the values of the work function of the holes in the geometrical configuration which we have postulated. The phenomenon of viscosity of liquids is explained on the basis of the quasi-crystalline structure of the liquid state and the thermal motion of particles with transference of momentum on collision. This represents the "hole" theory of viscosity. Writing  $A$  to represent the work function for the holes per gram molecule of liquid, the viscosity is given by

$$\eta = B \frac{T}{T_m} \cdot \frac{A}{RT} \quad (1)$$

where  $\eta$  is the viscosity coefficient,  $T$  and  $T_m$  the absolute temperatures ( $T_m$  melting temperature) and

$$B = 0.45 \frac{RT_m}{V_m} \sqrt{\frac{m}{\sigma}}$$

where  $\sigma$  is the surface tension and  $R$  the gas constant.

If, therefore,  $\eta$  and  $\eta'$  are the values of viscosity at two different temperatures  $T$  and  $T'$

$$A = R \frac{T T'}{T' - T} \log \frac{\eta T'}{\eta' T} \quad (2)$$

for a gram molecule of liquid. We assume the same type of structure in the liquid state under normal condition, and consequently the same work function in determining the increase in the number of holes when sound waves are incident upon the

liquid. It is apparent that if the sound waves do not possess the requisite amount of energy, no increase in the number of holes will result. While, if the frequency of the sound wave is large, the same result will again follow.

Table I gives the value of  $A$  for various liquids from the viscosity data as calculated from formula (3). Viscosities at various temperatures have been taken from *International Critical Tables*<sup>6</sup> and *Physical & Chemical Constants* by Kaye and Laby<sup>7</sup>.

Table II shows the values of the internal structural specific heat  $C_i$  as calculated from the formula

$$C_i = R \left( \frac{A}{RT} \right)^2 - \frac{A}{RT}$$

where  $A$  and  $R$  are as usual work function and gas constant respectively.

In applying the reaction rate theory, we assume that the excited condition is represented by the creation of holes in the liquid; Bauer's formula<sup>8</sup> (equation 14) leads to the result

$$\frac{d}{N^2} = \pi \frac{C'}{C^2} \frac{\gamma - 1}{C_p} \frac{C_i}{N_m} \left\{ 1 + \left( \frac{N}{N_m} \right)^2 \right\} \quad (3)$$

where  $d$  is the absorption coefficient,  $N$  is the frequency of sound waves  $C^1$  is the velocity of sound when ultrasonic absorption takes place and  $C^2$  the same without absorption,  $\gamma$  the ratio of specific heats,  $C_p$  specific heat at constant pressure,  $C_i$  the

TABLE I

Liquid	$A$ cal./mole
Carbon tetrachloride	3330
Benzene	3148
Chlorobenzene	2771
Chloroform	2201
Acetone	2250
Toluene	2207
Ethyl bromide	1812
Carbon disulphide	1802

TABLE II

Liquid	cal. deg. <sup>-1</sup> mole <sup>-1</sup>
Carbon disulphide	0.8937
Benzene	0.2769
Carbon tetrachloride	0.2207
Chloroform	0.6446
Chlorobenzene	0.4295
Toluene	0.9181
Acetone	0.6486
Ethyl bromide	0.8640
Acetic acid	0.2423

internal structural specific heat and  $N_m$  the dispersion frequency.

The maximum absorption per wavelength at  $N = N_m$  is given by

$$2\mu_m = \pi \frac{\gamma'}{\gamma} \frac{\gamma - 1}{C_p} C_i \quad (4)$$

where  $\gamma'$  is the ratio of specific heats when ultrasonic absorption takes place and  $\gamma$  the same without absorption.

The dispersion frequencies have been calculated from the relation

$$N_m = \frac{2\mu_m}{C} \left[ \frac{1}{d/N^2} \right] N \ll N_m \quad (5)$$

The dispersion frequencies have been determined for different liquids by substituting the experimental values of  $\frac{d}{N^2}$ .

These results have been recorded in Table III. The values of absorption and the sound velocity  $c$  as shown in columns 2 and 3 of Table III have been taken from Bauer's paper<sup>8</sup> (TABLE I) and that of acetic acid from Bazulin<sup>9</sup> and Pinkerton<sup>10</sup>. It is to be noted that a very small change in the values of viscosity at a particular temperature has a large effect upon the specific

heat  $C_i$  and consequently upon the maximum absorption frequency.

The experimentally determined value of maximum dispersion frequency in the case of acetic acid is 2.5 Mc. while the hole theory gives 4.87 Mc. If we assume that there are vibrational levels in addition, the requisite value of  $C_i$  comes out to be 0.246 cal. deg<sup>-1</sup> mole<sup>-1</sup> and  $A$  is 7211 cal./mole. Subtracting from this the hole energy 3291 cal./mole, we get 3920 cal./mole — the additional vibrational energy corresponding to the frequency 1382 cm.<sup>-1</sup>. This corresponds to frequency  $1381 \pm 5$  cm.<sup>-1</sup> for monomer acetic acid<sup>11</sup>.

It seems, therefore, that the energy levels of hole may have quantized values as suggested by Auluck and Kothari in their paper<sup>5</sup> "that the discrete energy levels of a hole may reveal their presence in the ultrasonic phenomenon".

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TABLE III

Liquid	$d/N^2 \times 10^{17}$ sec. <sup>2</sup> cm. <sup>-1</sup>	$c \times 10^4$ cm./sec.	$2\mu_m$	$N_m$ (Mc.)
Carbon disulphide	10,800	1.14	0.2789	23
Benzene	830	1.31	0.0979	90
Carbon tetra- chloride	513	0.91	0.0352	140
Chloroform	380	0.987	0.1806	480
Chlorobenzene	123	1.302	0.1540	983
Toluene	84	1.31	0.2278	2070
Acetone	60	1.17	0.2301	3278
Ethyl bromide	56	0.932	0.2608	5067
Acetic acid	12,000	1.2	0.0701	4.87

# Evaluation of Red Lead Pigments: Part III—Autocatalysis in Paint Sedimentation

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THE object of this paper is to report an interesting case of autocatalytic behaviour exhibited by a sample of "superfine" red lead paint during sedimentation. Sedimentation studies with a view to determine the particle-size distributions in red lead pigments were already reported by the author in an earlier paper<sup>1</sup>. Numerous samples of "ordinary", "non-setting", "high-disperse" and "superfine" samples of red lead were examined in the course of those investigations, both in the presence and also in the absence of an added dispersing agent such as aluminium stearate. A typical sedimentation curve and the size-distribution diagram deduced from the above by the graphical method of Svedberg

are shown in Figs. 1a and 1b. All the samples behaved in the normal way in respect of the sedimentation velocity. The "superfine" red lead sample, however, when made into a paint paste with 0.75 per cent of aluminium stearate in the usual mixture of raw and double-boiled linseed oils and dispersed in white spirit for the purpose of determining the rate of sedimentation, was found to give a curve which was distinctly S-shaped and which called for an explanation.

The autocatalytic behaviour disappeared when the paint dispersion was thoroughly shaken in the same sedimentation tube after 24 hr. and the experiment repeated. Figs. 2, 3 and 4 show the sedimentation curves for "superfine" red lead paint in the

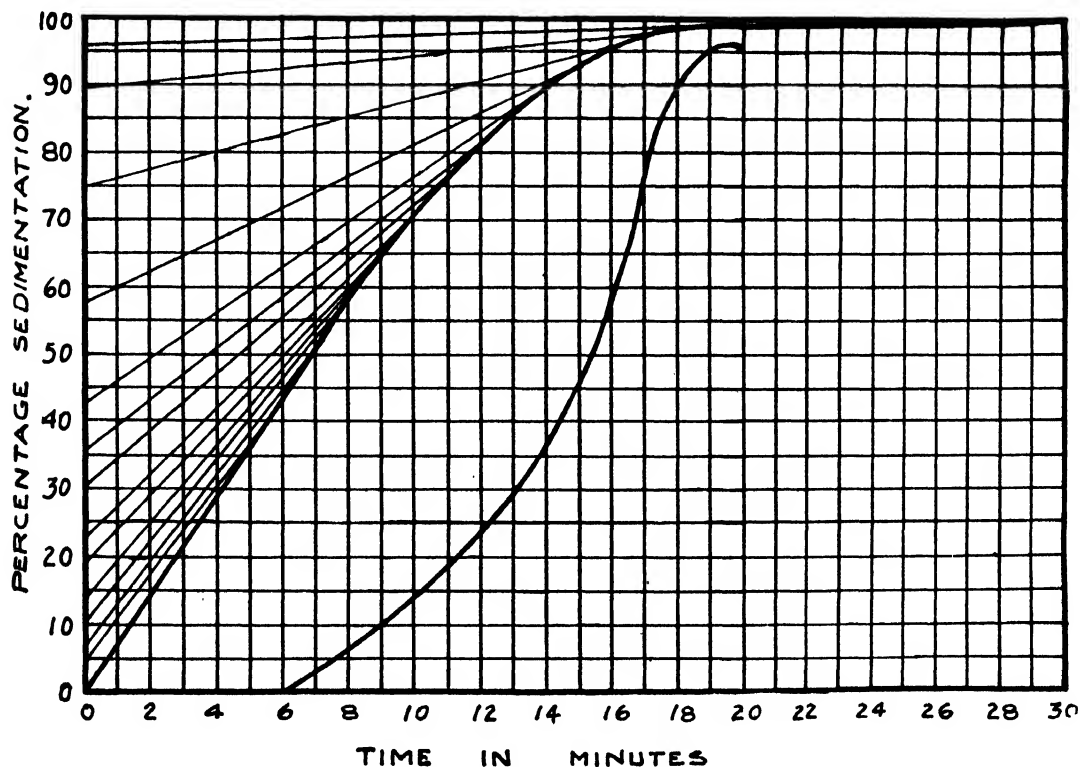


FIG. 1a — SEDIMENTATION CURVE, TYPICAL OF THE NUMEROUS SAMPLES OF RED LEAD.

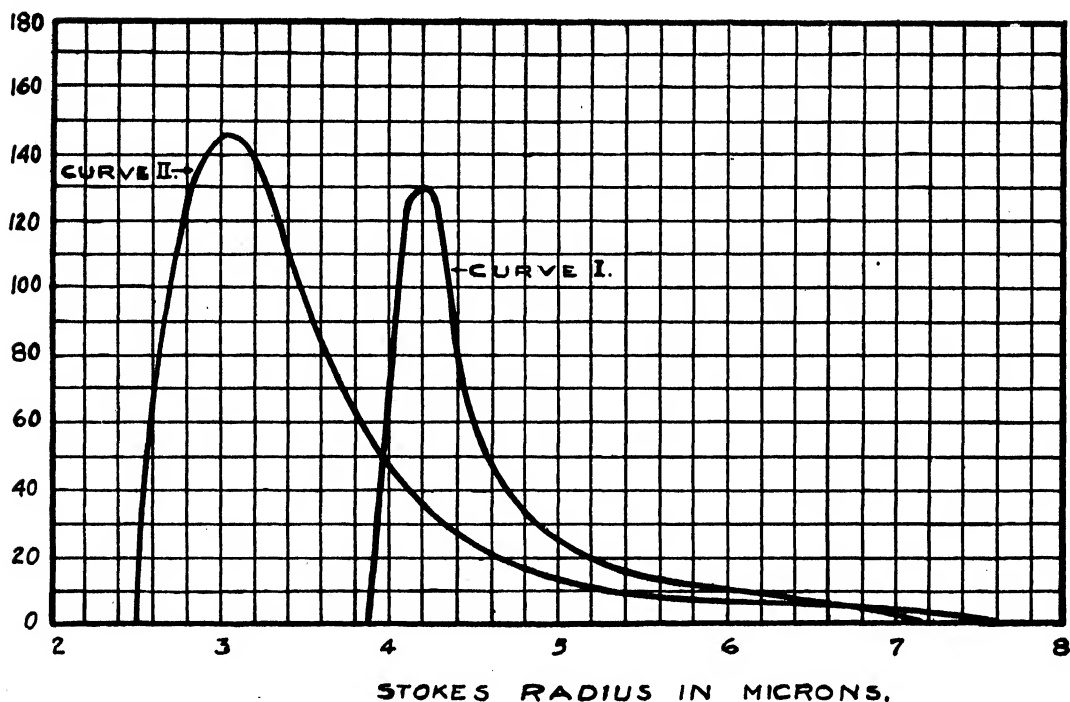


FIG. 1b — CURVE I : SIZE-DISTRIBUTION DIAGRAM, DEDUCED FROM ABOVE. CURVE II : SIZE DISTRIBUTION DIAGRAM FOR THE SAME PAINT, BUT WITH ALUMINIUM STEARATE AS A DISPERSING AGENT.

absence of aluminium stearate, in the presence of aluminium stearate and in the presence of the same when the experiment was repeated after 24 hr. in the same tube, respectively. The amount of aluminium stearate used was 0.75 per cent of the weight of the pigment.

The experiments were repeated in order to make sure that the autocatalysis was genuine and did not arise due to any flaw in the experimental procedure adopted, and the results were confirmed. None of the other samples so far examined exhibited this type of behaviour. The appearance of autocatalysis (FIG. 3) and its disappearance when the same system was examined after 24 hr. was somewhat curious. In order to track down the cause of such a behaviour, further experiments were carried out using fresh quantities of the same sample of red lead and repeating the experiment on the same system in the same sedimentation tube by shaking it vigorously at stated intervals of time before determining the rates of sedimentation. This gave interesting results which made it possible to offer a fairly satisfactory explanation regarding the causes

for the autocatalysis and the latter's significance.

#### Experimental Results

The apparatus used, the general experimental procedure and the method of calculation of the rates of sedimentation and the particle-size distribution diagrams were described in an earlier paper<sup>1</sup>. The sedimentation curve shown in Fig. 1 is typical of those obtained for numerous samples of red lead pigment of different qualities and description either when used as such or after incorporation of a small percentage of aluminium stearate. The "superfine" sample itself, without aluminium stearate, gave the same type of curve that is normally obtained, as can be seen from Fig. 2. It was the incorporation of 0.75 per cent of aluminium stearate, along with the "superfine" red lead while making the paint, that changed the character of the sedimentation, as will be seen from the distinctly S shape of the curves in the initial stages of sedimentation (FIG. 3). When observations were taken on the same paint system after a lapse of 24 hr. by shaking the tube for thorough

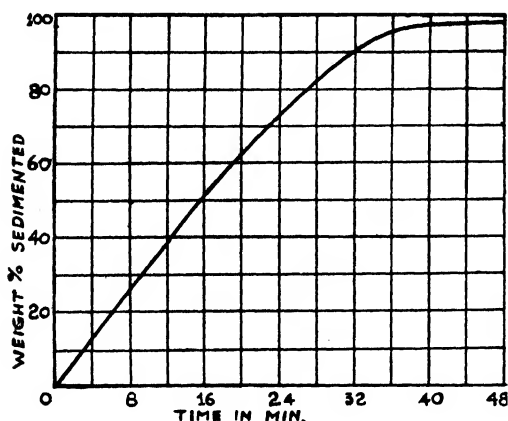


FIG. 2—SEDIMENTATION CURVE FOR "SUPERFINE" RED LEAD MADE INTO PAINT WITHOUT ALUMINIUM STEARATE.

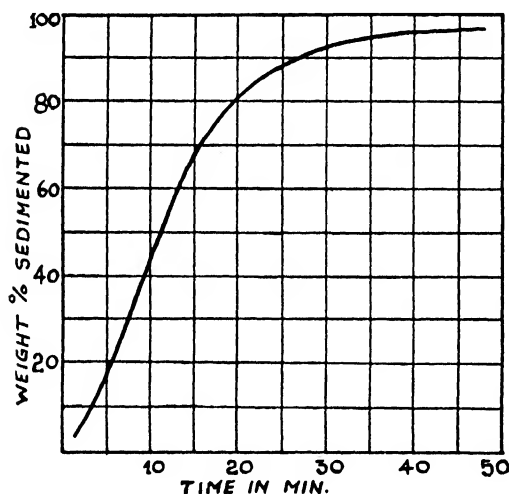


FIG. 3—SEDIMENTATION CURVE FOR THE SAME SAMPLE WITH 0.75 PER CENT ALUMINIUM STEARATE.

re-dispersion and noting the rates of fall, the autocatalytic behaviour disappeared altogether as will be seen from the sedimentation curve shown in Fig. 4. Similar experiments were repeated, starting with fresh samples of "superfine" pigment made into paint with the usual mixture of raw and double-boiled linseed oil and 0.75 per cent aluminium stearate, and dispersing in white spirit. The only important difference was that after the formation of the paint and dispersing in white spirit, instead of setting the systems aside for 24 hr. before taking the observations, they were set aside for stated intervals of time, viz. 20 min., 1 hr., 6 hr., and 47 hr., and the rates of sedimentation were determined. These sedimentation curves, espe-

cially for the initial stages of settling, are shown in Fig. 5. It will be seen from the curves that the autocatalytic behaviour which characterizes this system tends to disappear gradually, as more and more time is allowed to elapse after the first formation of the paint. The sedimentation curve for the 6 hr. interval system actually shows only a slight inflexion. The sedimentation curves for the same system, determined after 24 hr. and 30 hr., showed the normal behaviour without disclosing autocatalysis or other novel feature. When the observations were made after a still longer time-interval, namely 47 hr., the rate of sedimentation again failed to conform to the normal type, and indicated the existence of

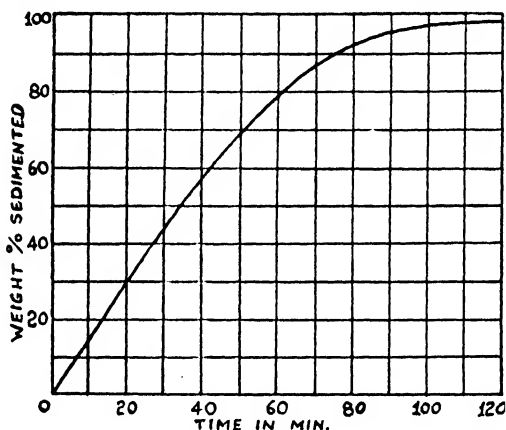


FIG. 4—SEDIMENTATION CURVE FOR THE SAME SYSTEM AS IN FIG. 3, BUT 24 HR. AFTER THE PREPARATION OF THE PAINT.

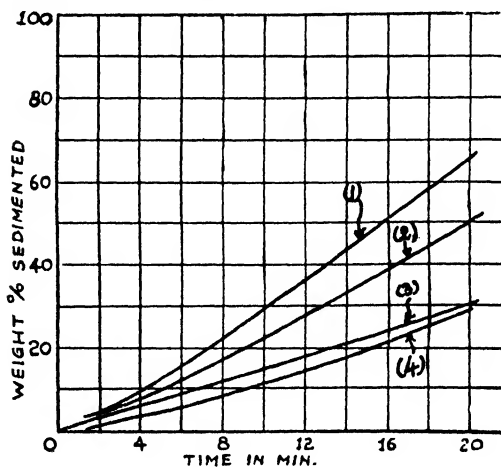


FIG. 5—SEDIMENTATION CURVES FOR THE SAME SYSTEM AS IN FIG. 3, BUT AFTER THE PREPARATION OF THE PAINT: (1) 20 MIN.; (2) 1 HR.; (3) 6 HR.; (4) 47 HR.



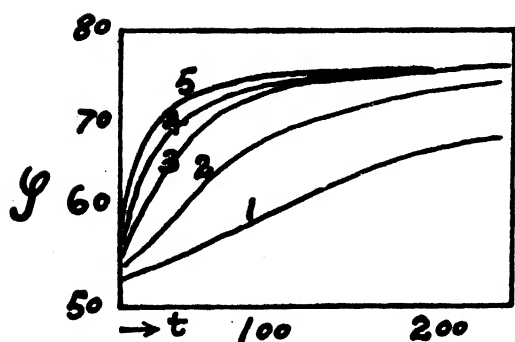


FIG. 6 — SHOWS COAGULATION VELOCITY CURVES OF  $Al_2O_3$  SOL BY KCl SOLUTION.

certain complicating factors arising from the precipitation of gels of lead soap, which could be distinctly noticed as thin flakes floating in the supernatant white spirit and almost forming a layer on the surface of the sedimented particles of red lead. In this connection it may be recalled that in the preparation of double-boiled linseed oil, sulphates, borates, oxides and other salts of lead, manganese, cobalt, cerium, etc. (driers), are added to the oil before heating. These bases form soaps by reaction with the fatty acids of the oil. These soaps, especially of lead, get separated out as gels either on standing or on dilution with thinners such as white spirit or turpentine. The disturbing effect of the flakes of gel on the normal course of sedimentation of the pigment particles so as to make it deviate from Stokes' law is, therefore, quite obvious and explains the type of curve obtained in the case of the system set aside for 47 hr., referred to above.

### Discussion

The curious nature of the sedimentation curves described above, which characterizes only the "superfine" red lead sample, seems difficult of explanation. The extreme fineness of the particle-size of this pigment, on careful consideration, appears to provide the necessary clue to a proper understanding of all the observed facts.

It is well-known that autocatalysis is a common feature in the "slow" coagulation of colloids. With increasing concentration of the coagulating electrolyte and the consequent change to "rapid" coagulation, the autocatalysis also gradually vanishes. Freundlich mentions a number of examples of autocatalytic behaviour, viz. the coagulation of aluminium oxide sol by potassium

chloride solution, the sedimentation of kaolin and barium sulphate suspensions, etc.<sup>3</sup> Figs. 6 and 7 (reproduced from Freundlich) clearly show the effects of increasing the concentration of the coagulating electrolyte on the coagulation velocity of aluminium oxide sol and on the rate of sedimentation of barium sulphate. At low concentrations of the coagulator, the curves are distinctly autocatalytic, but as the concentration is gradually increased, the initial S shape of the curves also likewise tends to vanish and when the coagulator-concentration is made sufficiently high, the curves tend to assume a straight-line shape. It is explained that in such cases the original primary particles form coarser flakes or aggregates under the influence of the coagulator, and that they settle more quickly only when the time up to their formation, namely the "aggregation time" of Oden, has elapsed.

The foregoing examples indicate that in dealing with paint systems of the type provided by "superfine" red lead, one is confronted with "colloid phenomena", the significance of which, in so far as it concerns the field of practical applications, needs careful elucidation, since it is not unlikely that it may lead to conclusions of far-reaching importance not easily apparent or understandable when considered in the orthodox manner of treatment.

In a previous paper<sup>3</sup> (Part II of the *Evaluation of Red Lead Pigments*), the author has drawn attention to the fact that the "standard specifications" are generally deficient in respect of the prescribed particle-size characteristics, and that one is liable to over-estimate the usefulness of a paint if he bases his judgement on the particle-size properties stated in the specifications. The

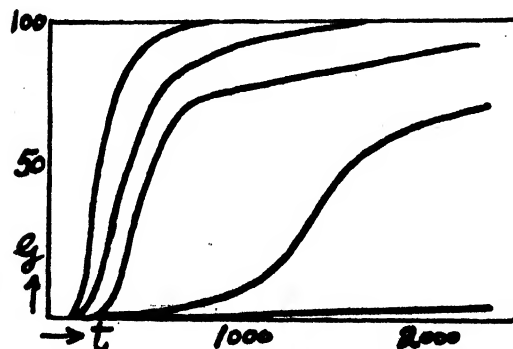


FIG. 7 — SHOWS SEDIMENTATION VELOCITIES OF  $BaSO_4$  SUSPENSION AT DIFFERENT CONCENTRATIONS OF AMM. NITRATE.

defect of the standard specifications seems to lie in the rough upper limit which alone is usually stated, without mentioning anything about the lower limits of the particle size. It is stated, for example, that not more than 1 per cent of the pigment should constitute particles retained on a 325-mesh sieve ( $44\ \mu$ ). This specification merely helps in ensuring that the pigment does not contain coarse particles of dirt, grit or other foreign matter. As will be seen from the particle-size data presented by the author<sup>1,4</sup>, none of the 15 samples examined indicates the presence of particles approaching 44 microns diameter and almost all of them are confined to the region below 14-16 microns diameter. While the conditions laid down in the specifications appear to be fulfilled in the case of *all* these pigments, it must be noted that the different samples possess properties widely varying in their practical usefulness such as covering power, non-setting nature, protecting power against corrosion, etc.

The importance and need for specifying a *lower* limit too becomes obvious from the significant behaviour shown by the "superfine" sample. Of all the samples, this one appears to be almost the best in respect of particle fineness, peak size, specific surface, dry bulk, oil-absorption, density, etc. One is, therefore, apt to consider this to be the most suitable and satisfactory pigment. In fact there is an almost universal over-emphasis on the particle-fineness desired, with the result that it is considered by most people that the finer the pigment, the more it is to be preferred. Actual exposure tests, on the contrary, have shown that this argument is only of limited validity. Appearance of rust on test panels painted with "superfine" red lead occurred much earlier than in the case of panels painted with a comparatively coarse pigment. Thin layers of small pigment particles are more readily affected by light than tinctorially equivalent layers of large particles<sup>5</sup>. For this reason alone, there appears to be adequate justification for fixing or specifying the "lower" limit of particle size.

A second consideration of far-reaching significance arises from the observations of autocatalysis recorded in this paper. Normally, the use of dispersing agents of the type of aluminium stearate may be advocated to counteract the "setting" tendency of paints to procure certain obvious advantages in the preparation and storage of the

so-called "non-setting" red lead paints. When this procedure is adopted in the case of the "superfine" type of samples, the effect will be to transform the paint into a near "colloidal" or actual colloidal system with all its logical consequences.

Ordinarily, the "dispersion stability" of these systems can be gauged from determinations of rates of sedimentation, under conditions of extreme dilution, by an inert solvent such as white spirit. In the case of paints comprising of near-colloidal systems, the inert diluent is no longer inert, but acts as a coagulator causing flocculation or aggregation. This is shown by the autocatalytic type of sedimentation curves obtained in "superfine" red lead plus aluminium stearate system. When the experimental method adopted, which is supposed to be valid only for systems consisting of particles freely falling in an infinite, inert medium in accordance to Stokes' law, is thus vitiated by flocculation or aggregate formation, it becomes difficult to draw quantitative conclusions as regards the particle-size characteristics.

Secondly, in the practical field as well, the presence of colloidal systems, easily liable to flocculation under actual conditions of application of the paints, very much detracts from the usefulness of the paint under question, as far as rust-prevention, hiding power, etc., are concerned. The "superfine" red lead paint, when slightly thinned and applied on the surface which it is desired to protect, will "coagulate" in the same manner as happens when it is suddenly diluted with white spirit and shaken in the sedimentation tube (as described in this paper). This will lead to segregation of the pigment particles in streaks or spots or in other irregular manner, causing a number of lacunae, devoid of the pigment layer, to appear on the painted surface. These "open" unprotected spaces will be the first to be attacked by agencies causing corrosion.

It will be seen from what has been stated above that the "quality" of pigments cannot be increased indefinitely by increasing the "fineness" of particles by continued or improved techniques of grinding or other means. Incidentally, the results laid down in this paper lend weight to the author's supposition stated in previous papers that "superfine" red lead is nothing but "orange lead" or "orange mineral" of very fine particle size, no doubt, but also, at the same time, of doubtful usefulness. The possibility

and the easy scope for the formation of these paints into near-colloidal systems will be easily understood if it is remembered that "orange mineral" is made not from massicot but from white lead "tailings" which form as a froth in the grinding and levigating operations, and is, hence, of an extremely soft, light and fine texture.

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## Evaluation of Red Lead Pigments : Part IV—Improvement of Dispersion Stability of the Paints

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**A**N assessment of the hard-setting tendency of red lead paints can be made normally by tests conducted in the storage container after different periods of storage. But this procedure involves long waiting before an opinion can be pronounced on the samples under test. It was pointed out in the previous papers<sup>1</sup> how the setting quality of a paint can be estimated by an examination of the dispersion stability. Measurement of the latter could be done by determining the rates of sedimentation of the paint particles under conditions aggravating the flocculation tendency brought about by extreme dilution with an inert solvent, namely white spirit in this case. The resistance of the pigment particles to sedimentation under these conditions gives an idea of the dispersion stability. Advantage was taken of this procedure in setting studies of numerous samples of red lead. A similar procedure was reported to have been adopted at the Paint Research Station, Teddington, in the evaluation of other pigments. The hypothetical particle-size distribution having the same rate of sedimentation as the pigment particles settling down under conditions of extreme dilution, as in the case of these experiments, would give a very useful picture of the pigment quality. In experiments previously reported (*loc. cit.*), the particle-size distributions in different types of red lead,

both as such and also when the paint was sought to be improved by the incorporation of a small percentage of aluminium stearate as a dispersion agent, indicated the great improvement conferred by aluminium stearate in respect of the natural setting tendency.

In this paper are reported the results of further studies involving the use of certain other dispersing agents. Protection of pigment particles, it has been pointed out, may arise either by the formation of an adherent envelope of long-chain polar molecules or by a cohesive envelope such as that produced by polymerized oils, not strongly polar in character and consequently not present as an absorbed layer on the pigment surface, but as a sort of coherent skin around the particles in the same manner as colloidal sols of gold are stabilized by gelatine and other protective agents. It will be seen that a careful examination of the size-distribution diagrams gives some insight into the mechanism of the anti-setting property conferred by different dispersing agents.

#### Experimental Results

All the experiments were conducted on the same sample of fairly satisfactory red lead pigment purchased from the market. In each experiment, 10 gm. of the pigment were formed into stiff paint with 3 c.c. of a mixture of raw and double-boiled linseed oil

(1:2 by volume), except where stated otherwise. The duration of mixing and the detailed procedure adopted in forming these paints were maintained the same throughout. The paint so formed was thinned to a volume of 300 c.c. using white spirit in the sedimentation tube (modified Kelly's tube) and the rates of sedimentation were determined by observing the fall of the meniscus in the manometric limb by means of a cathetometer. Calculation of the particle-size distributions was made by the graphical method employed by Svedberg.

The results obtained are shown in Figs. 1 to 10. The description of the paint system in the case of each experiment, the peak radius, the size range and the type of size distribution are indicated in Table I. There are different ways of expressing the particle size of heterogeneous or polydisperse mate-

rials by a single figure. The usual types of diameters referred to by statisticians are the mean particle diameter,  $d_1$  or  $d_{me}$ , the median particle diameter,  $d_{med}$ , the Modal particle diameter,  $d_m$ , the length average, the mean volume-surface diameter, the weight mean diameter, the mean surface diameter, the mean volume diameter, etc. Of these, the most frequently used averages are the first three only. The first one, namely  $d_{me}$ , as its name implies, is only the arithmetical average diameter given by the expression

$$d_{me} = \frac{\sum nd}{\sum n}$$

The second,  $d_{med}$ , represents the diameter of a particle so chosen that the number of particles of larger diameter is the same as those of smaller diameter. The third,  $d_m$ , the Modal particle diameter, represents the

TABLE I

No.	DESCRIPTION OF PAINT SYSTEM	PEAK RADIUS microns	RANGE microns	TYPE OF DISTRIBUTION	REF. TO FIG.
1	No dispersing agent	4.0	3.6	Slight left-skew	1
2	With 0.25% Al. stearate	4.7	3.6-2	Symmetrical	2
3	With 0.5% Al. stearate	3.6	2.5-6	Nearly symmetrical	3
4	With 0.75% Al. stearate	3.7	2.5-5.8	Symmetrical	4
5	With stand oil + raw linseed oil	4.25	3.6	do	5
6	With stand oil alone	3.6	2.5-6	do	6
7	Repetition of 6, with fresh pigment sample	3.6	2.8-6.5	Slight right-skew	7
8	With 2% plaster of Paris	4.3	3.6	Nearly symmetrical	8
9	With 5% plaster of Paris	3.7	2.6	Symmetrical	9
10	With oleic acid + raw linseed oil	5.3	3.2-7.5	do	10

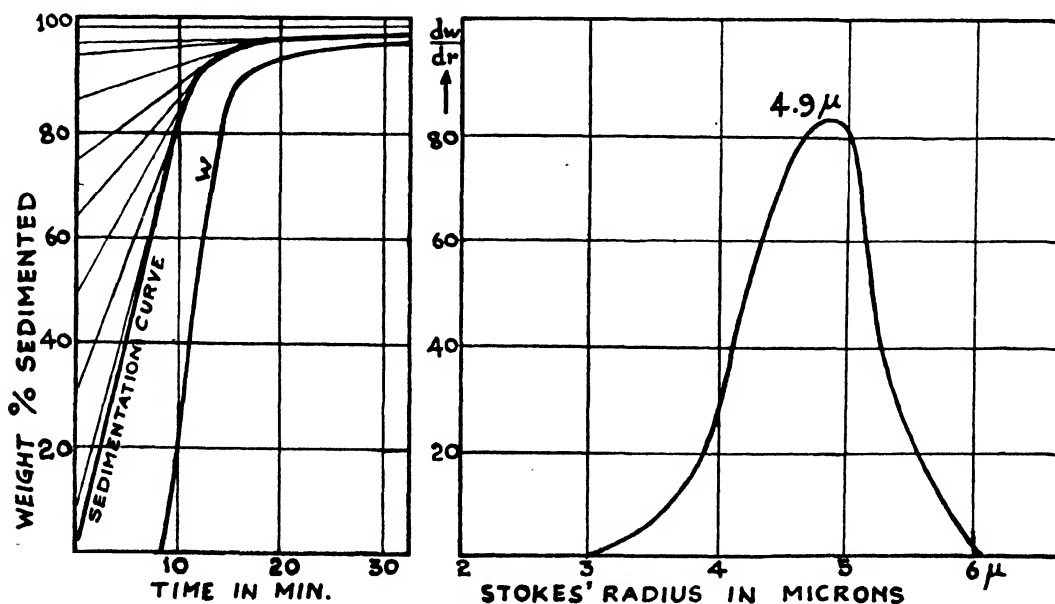


FIG. 1—SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM USING SVEDBERG'S GRAPHICAL CALCULATION METHOD; RED LEAD WITHOUT ANY DISPERSING AGENT.

most probable diameter for a particle in a mixture of many sizes, that is to say, that it is the diameter of the largest number of particles. It is considered that an idea of  $d_m$  will be the most significant in the studies reported in this paper. The value of  $d_m$  is given by the peak of the size-distribution diagram and in Table I, the peak radius is,

therefore, the Modal particle radius. This is also the same as the arithmetical mean particle diameter  $d_{me}$  or the median diameter  $d_{med}$ , in cases where the size-distribution is symmetrical.

The first 4 experiments reveal progressive improvement in respect of dispersion stability, brought about by increasing use of

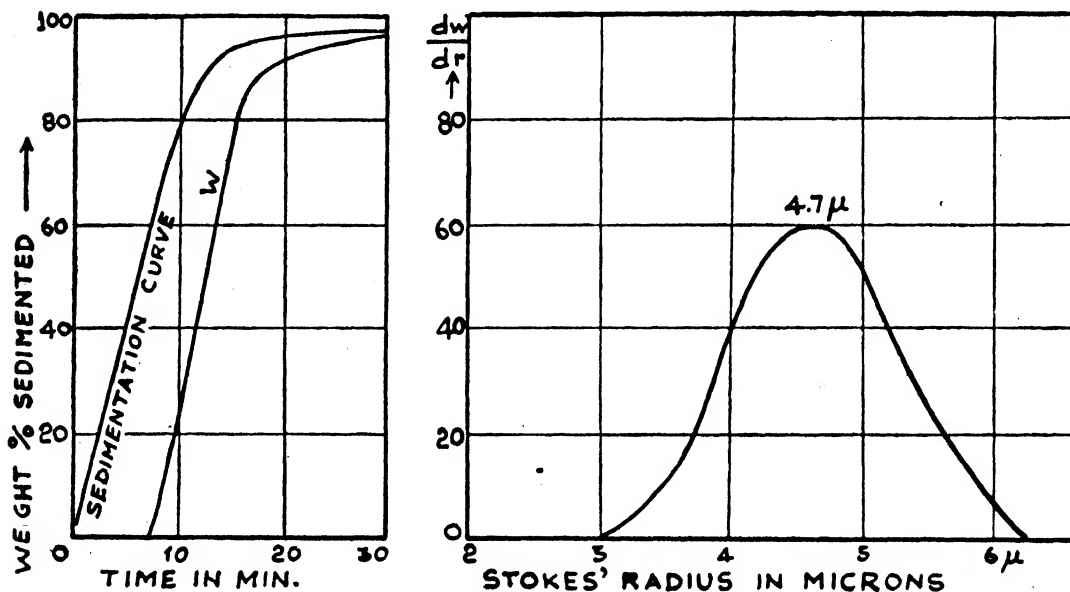


FIG. 2 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM; RED LEAD + 0.25 PER CENT ALUMINIUM STEARATE.

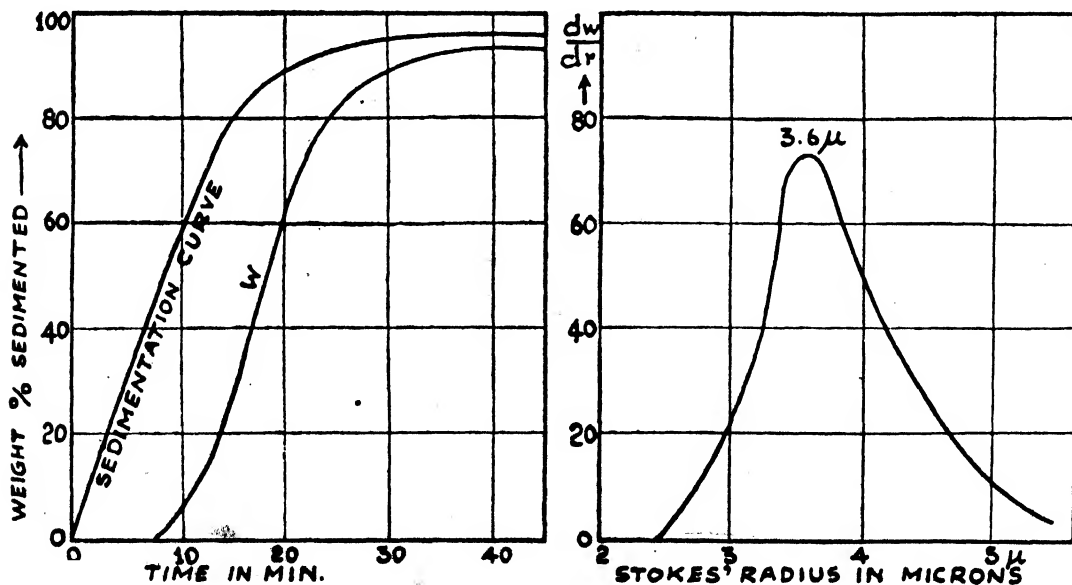


FIG. 3 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM; RED LEAD + 0.5 PER CENT ALUMINIUM STEARATE.

aluminium stearate as a dispersing agent. This is clearly brought out by the progressive decrease of the peak radius given in column 3 and also by the narrowing down of the size range given in column 4 as one proceeds from experiments 1 to 4 ( Figs. 1 to 4 ). 0.5 per cent or 0.75 per cent of aluminium stearate on the weight of the pigment seems

to be the optimum for securing the best dispersion stability.

In experiments 5, 6 and 7 ( Figs. 5 to 7 ), the effect of using stand oil, either in partial or total replacement of the linseed oil usually employed, is shown. In experiment 5, 10 gm. of the pigment under test were mixed with 1.5 c.c. of raw linseed oil and 1.5 c.c. of

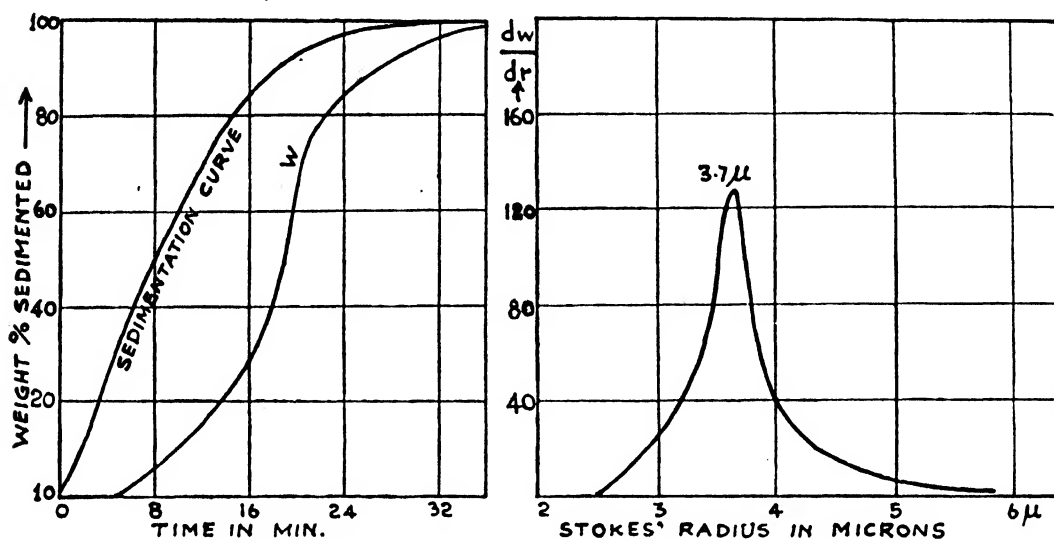


FIG. 4 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM ; RED LEAD + 0.75 PER CENT ALUMINIUM STEARATE.

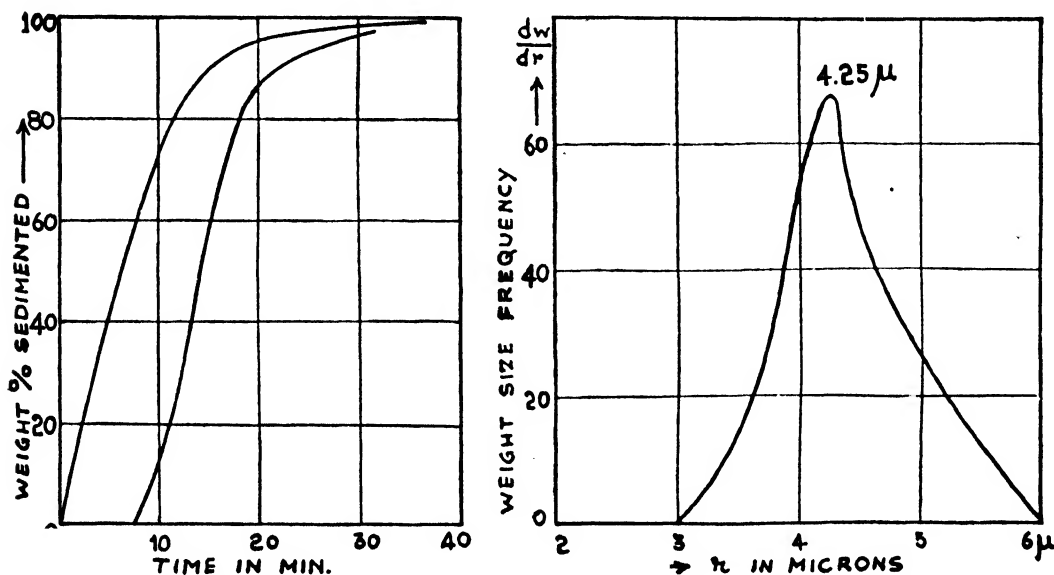


FIG. 5 — SEDIMENTATION CURVE AND PARTICLE-SIZE DISTRIBUTION DIAGRAM ; SAME SAMPLE OF RED LEAD BUT HALF OF THE 30 PER CENT ( BY VOLUME ) OF RAW AND DOUBLE-BOILED LINSEED OIL REPLACED BY AN EQUAL VOLUME OF STAND OIL, THE OTHER HALF BEING RAW LINSEED OIL.

stand oil for forming the paint. In experiment 6, no linseed oil was employed, and instead, 3 c.c. of stand oil alone were used. Experiment 7 is a repetition of experiment 6 using 3 c.c. of stand oil with a fresh batch of pigment drawn from the same original stock. This experiment indicates the extent of reproducibility of the results. In experiments 8 and 9 (Figs. 8 and 9), the paints were formed by using 2 per cent and 5 per cent,

by weight, of plaster of Paris respectively. The usual mixture of raw and double-boiled linseed oils (1 : 2 by volume), employed in the case of experiments 1 to 4, was used in forming the paint. The plaster of Paris was pre-heated for 4 hr. at 125°C. before incorporation in the paint. In experiment 10, 10 gm. of the pigment was mixed with 1.5 c.c. of oleic acid and 1.5 c.c. of raw linseed oil. It will be seen from the distribu-

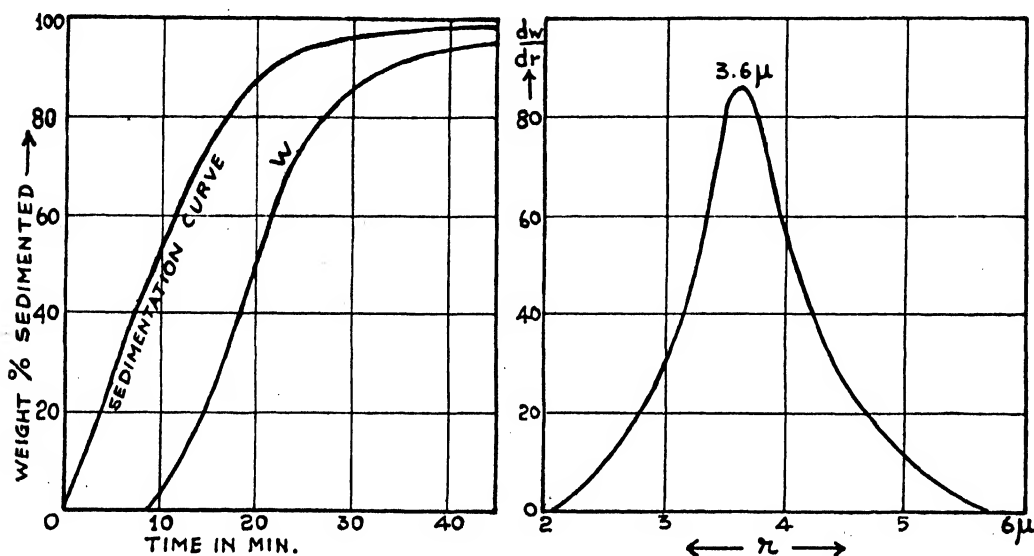


FIG. 6 — RED LEAD MADE INTO PAINT WITH AN EQUAL VOLUME OF STAND OIL IN TOTAL REPLACEMENT OF 30 PER CENT (BY VOLUME) OF LINSEED OIL.

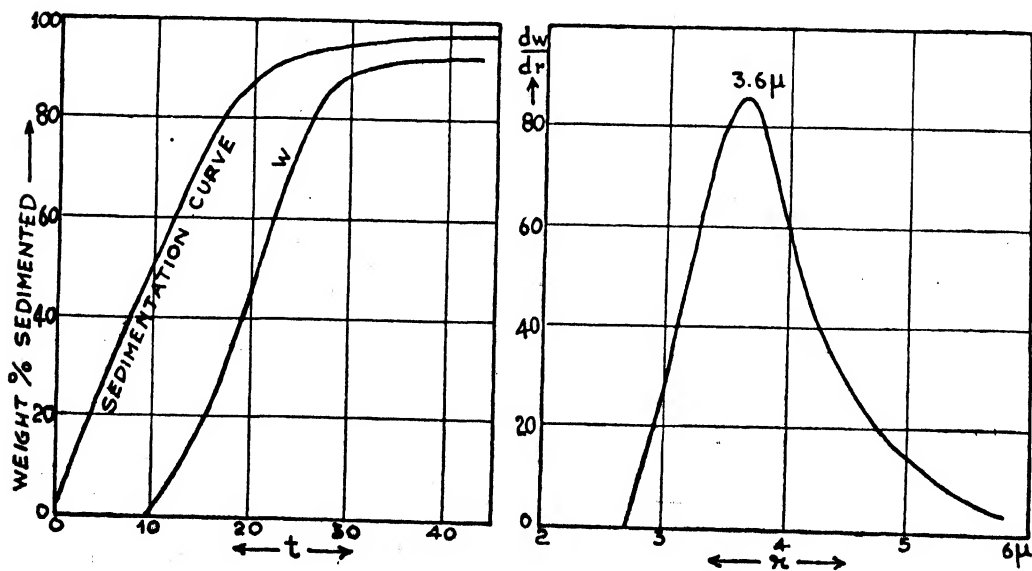


FIG. 7 — SAME AS IN FIG. 6, BUT WITH A FRESH BATCH OF PIGMENT.

tion diagram (FIG. 10) that far from acting as a dispersing agent, oleic acid has actually reduced the dispersion stability thereby leading to more rapid setting. The peak radius as well as the size range (cols. 3 and 4) are higher in this case than the values for experiment 1 where the pure test sample as such was employed without any dispersing agent.

It may be concluded from the results given above that if the red lead paints are formed by total replacement of the usual linseed oil mixture by stand oil, the quality of the paints in respect of setting tendency and dispersion stability is improved to the same extent as can be secured by the incorporation of an optimum percentage of aluminium stearate. The same may be

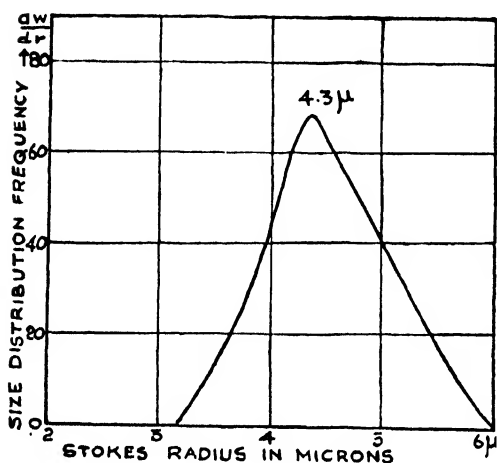


FIG. 8 — RED LEAD WITH 2 PER CENT PLASTER OF PARIS.

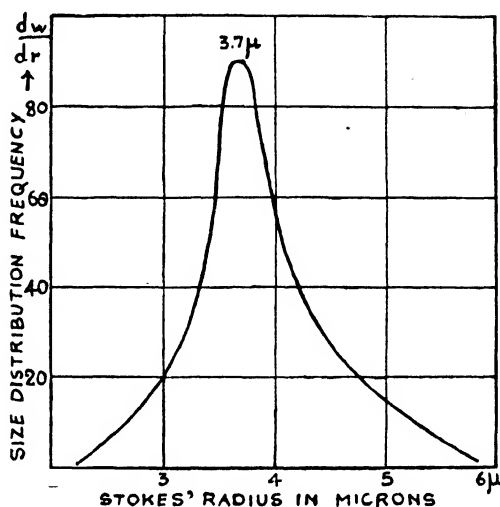


FIG. 9 — RED LEAD WITH 5 PER CENT PLASTER OF PARIS.

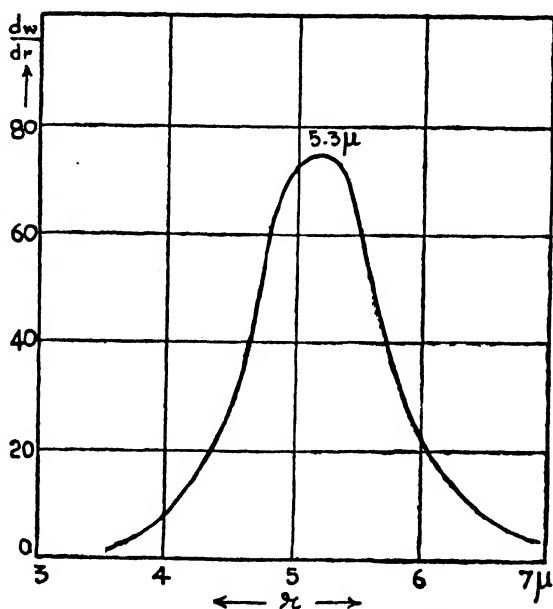
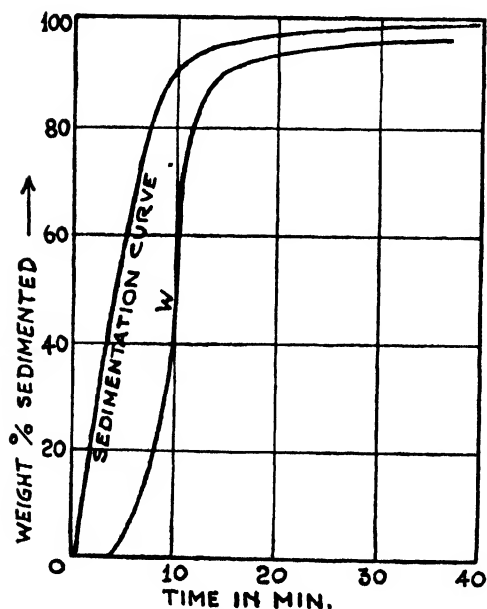


FIG. 10 — RED LEAD; HALF OF THE 30 PER CENT (BY VOLUME) OF LINSEED OIL IS SUBSTITUTED BY AN EQUAL VOLUME OF OLEIC ACID AND THE REMAINING HALF BEING RAW LINSEED OIL.



said about the use of plaster of Paris (5 per cent by weight) on the pigment sample. A few experiments carried out using talc, barytes and other substances as extenders or suspending agents gave unsatisfactory results. Although some of them appeared to slightly retard the setting, the results obtained were not considered satisfactory for extended investigations. As a matter

of fact, barytes increased the *settling* tendency which is also an undesirable feature for any paint sample.

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## Electrolytic Reduction of *ortho*-Nitrochlorobenzene to *meta*-Chlor-*p*-Aminophenol

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THE electro-reduction of *o*-nitrochlorobenzene in acid emulsion to *meta*-chlor-*p*-aminophenol does not appear so far to have been reported in literature. Indeed, no reference to the electro-synthesis of halogen-substituted aminophenols is found in literature and investigations even on the chemical methods of their preparation appear to be scarce. This accounts partly for the general lack of recognition of such compounds as possible dyestuff intermediates. Apart from their value as potential dye-intermediates, a study of these compounds would be of considerable interest in throwing light on the influence of such halogen substitution on the stabilization of the molecule against oxidative changes thereby conferring on them better keeping qualities. This is a factor of not little importance in making them suitable for employment as photographic developers in tropical climates.

*m*-Chlor-*p*-aminophenol was first described in a *D.R.P.*<sup>1</sup> as the product of reduction of 3-chlor-4-nitrophenol by iron and hydrochloric acid at 80°-100°C. and the m.p. was given as 160°C. It was also mentioned as a possible photographic developer. Later, Hurst and Thorpe<sup>2</sup>, in the course of their work on the preparation of phenacetin by the reduction of *p*-nitrophenetole using tin and conc. hydrochloric acid, obtained a chloro-phenetidine and its acetyl derivative

was de-ethylated by them in the presence of aluminium chloride to a product which was identified as *m*-chlor-*p*-acetyl-aminophenol (crystallized from chloroform, m.p. 121°C.). Hodgson and Kershaw<sup>3</sup> had also prepared this compound by the reduction of 3-chloro-4-nitrophenol in alkaline solution using sodium hyposulphite for the purpose (m.p. 160°C.; N-acetyl derivative, m.p. 121°C.). More recently, Kiewict and Stephen<sup>4</sup> obtained the diacetyl derivative of the compound (3-chloro-4-acetamido-phenyl acetate, m.p., 127°C.) by reducing 3-chloro-4-nitrophenol in acetic anhydride with solid stannous chloride. In all these cases, the products have been obtained by devious means and the yields too have not been specified.

When *o*-nitrochlorobenzene is subjected to cathodic reduction in strongly acid media, according to accepted principles, the initially formed  $\beta$ -chloro-phenyl hydroxylamine undergoes isomerization in part to the corresponding aminophenol, viz. *m*-chlor-*p*-aminophenol and also reduction in part to *o*-chlor-aniline, the relative proportions of the two depending upon the conditions of the experiment. Previous experience in regard to the preparation of aminophenolic derivatives by electro-methods had shown that these conditions which determine the maximum formation of the aminophenolic body are mainly concerned with the employment of suitable cathodic material,

either an alloy or simple metal, in conjunction with catalysts which suppress amine formation and simultaneously help the speed of isomerization of the hydroxyl-amino compound. Apart from these primary considerations, other factors like strength of acid, temperature of reduction, current density and depolarizer concentration also play a part in determining the nature and extent of the reduction. These latter factors, however, have been to some extent standardized, so that the experiments in the reduction of *o*-nitrochlorobenzene in acid medium were conducted only under the most favourable conditions determined previously, and were confined mainly to the study of the influence of variations in cathodic material and catalysts on the yields of the products.

### Experimental

The experimental procedure involved in both the reduction and isolation of the products in this investigation is similar to that worked out in connection with that of *o*-nitro-toluene<sup>5</sup>. One exception, however, has been in the use of a solvent like xylene since only by its employment the depolarizer could be brought to the top of the catholyte, thereby facilitating agitation and subsequent emulsification which otherwise would have been hampered, owing to the denseness of the nitrochlorobenzene and its consequent tendency to settle to the bottom in spite of vigorous agitation of the mass. The use of a volatile solvent, especially in an open cell, made it necessary to maintain the temperature of reduction rather low (about 50°-55°C.), consistent with efficient working, so as to reduce loss. Moreover, it was found that neither a higher temperature nor the passage of current in excess of that required by theory helped to improve the yield of the products. Only the theoretical amount of current, viz. 64 amp./hr. was, therefore, employed for the reduction.

As regards cathodic material, monel and copper (pure and amalgamated), and lead were employed in this study. Copper was found to be the most suitable in providing the maximum yield of the chlor-aminophenol (29.1 per cent of theory) and lead for the chloraniline (34.6 per cent of theory), in conjunction with copper sulphate as catalyst. Amalgamation of copper did not improve the yield of the aminophenol, although the current efficiency rose from

54 per cent to 65 per cent. Apart from copper sulphate, other catalysts such as sulphates of mercury, calcium, zinc and ammonium and iodide of potassium were also tried. A low yield (10 per cent) of aminophenol resulted when mercuric sulphate was used in the presence of copper cathode, while the rest of the catalysts were to be found to be quite unsatisfactory. Calcium and ammonium sulphates were employed as a modification of the suggestion by Brand Strache<sup>6</sup> who found a mixture of the chlorides to be effective in the chemical reduction of nitrobenzene to phenyl-hydroxylamine. According to Khirkhof<sup>7</sup> presence of iodides in the catholyte greatly increased the speed and efficiency of reduction of nitro compounds by acid electrolysis, but we are unable to confirm this in the present case.

The optimum conditions worked out and the results obtained so far for the preparation of *m*-chlor-*p*-aminophenol by acid electrolysis of *o*-nitrochlorobenzene are given below:

**Conditions** — Cathode: copper; catholyte: 100 gm. of *o*-nitrochlorobenzene dissolved in 100 c.c. of xylene and kept in 400 c.c. of 30 per cent sulphuric acid; anode: lead; anolyte: 50 c.c. of 30 per cent sulphuric acid; catalyst: 10 gm. of copper sulphate; temperature: 50°-55°C.; current strength: 20 amp./hr.; current density: 5.99 amp./sq. dm.; total current passed: 64 amp. hr. (theoretical).

**Results** — Current efficiency: 54 per cent; *o*-chlor-aniline: 24 per cent of theory; *m*-chlor-*p*-aminophenol: 29.1 per cent of theory, m.p., 160°C.; diacetyl derivative, double crystallized from alcohol and from chloroform, m.p., 131°C.; reduces ammoniacal silver nitrate to a fine mirror of silver and colours ferric chloride violet.

### Discussion

The amounts of chloraniline and chlor-aminophenol account for only a little over half (28.7 gm.) of the nitrochlorobenzene reduced. On titrimetric analysis of the mother liquor for the aminophenol remaining in solution, according to a modification of Brigham and Lukens' method<sup>8</sup>, another 8.8 gm. of the used up depolarizer could be accounted for. Even after this there still remained about 30 per cent of the reduced

nitro compound to be accounted in terms of converted products.

Although it is known that in a not too strongly acid medium a suspension of a nitro compound, when electrolytically reduced, yields, besides an amine and aminophenol, azo and azoxy benzenes and even benzidine<sup>9</sup>, these side reactions are known to occur only slowly<sup>10</sup> and cannot, therefore, account for all the 30 per cent of the nitro compound reduced, nor can losses due to evaporation explain this. Since it is very probable that an equivalent amount of the hydroxyl-amino compound had been formed, a plausible explanation would be found in the assumption that it remained as such in solution, not having a chance, under the conditions of the experiment, either to be isomerized to the aminophenol owing to steric hindrance or to get reduced further to the amine. It, therefore, becomes necessary not only to study the course of the reaction under different conditions but also to identify the various products formed before any attempt is made to improve the

yields of the aminophenol. With this end in view, a set of experiments is planned and details will be published separately.

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## Miscibility of Castor Oil with Some Petroleum Products

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TESTS for the purity of castor oil such as the determination of the Frabot Number<sup>1</sup> and Benzine point<sup>2</sup> have been based on its partial miscibility with petroleum ether. The *British Pharmacopoeia*<sup>3</sup>, for identification of castor oil, states that it "mixes completely with half its volume of light petroleum (B.P. 50° to 60°C.), and is only partially soluble in two volumes". Atkins<sup>4</sup> studied critical solution temperatures of castor oil with petrols of different sources and observed that the presence of aromatics and naphthenes lowers the miscibility temperatures of the paraffins present. The values obtained by different

workers for the critical solution temperature have differed mainly on account of the uncertain composition of the petroleum fractions used, and the wide boiling ranges of these fractions.

There is a general impression that castor oil and petroleum ether are immiscible at room temperatures usually prevalent (c. 25°-30°C.). This is not entirely correct since the two mix completely at certain volume ratios, irrespective of the type of the petroleum ether<sup>5</sup>. The present work was undertaken with a view to studying the behaviour of petroleum ether-castor oil binary mixtures over the whole

composition range. In addition, the miscibility of castor oil with kerosene, technical white oils and lubricating oils has also been studied.

### Experimental

**Materials Used** — Castor oil: pure cold-drawn filtered oil with the following characteristics was used:

Sp. gr. at 29.5°/29.5°C. 0.9557

Ref. index at 29.2°C. 1.4780

Acid value, 1.9

Sap. value, 183.0

Hydroxyl value, 158.3

The solvents used in this study are given in Tables I and II.

**Procedure** — Mixtures of castor oil and the various solvents were made on volume basis and their turbidity temperatures were determined in the usual way. Mixtures which were clear and homogeneous at room temperature were cooled down gradually in a water bath and the temperature at which faint turbidity appeared was noted. Mixtures which were immiscible were heated gradually in the water bath until they were homogeneous and then were allowed to cool down slowly until faint turbidity appeared.

TABLE I

SOLVENT	QUALITY	BOILING RANGE, °C.	ANILINE POINT, °C.
Petroleum ether A	C.P.	98-118	53.7
Petroleum ether B	C.P.	87-94	57.2
Petroleum ether C	C.P.	65-70	58.1
Kerosene	Commercial	170-250	71.1

TABLE II

SOLVENT	QUALITY	SP. GR. AT 30°/30°C.	REF. INDEX AT 29.5°C.	VISCOSITY IN CENTIPOISES AT 35°C.	ANILINE POINT °C.
White oil I	Technical	0.8593	1.4727	14.59	97.5
White oil II	Technical	0.8653	1.4753	12.46	91.3
Shell BD <sub>2</sub> oil	Lubricating	...	...	...	82.2

TABLE III

CASTOR OIL VOL. %	PETROLEUM ETHER C (65°-70°C.)	
	Turbidity temperature, °C.	Clearance temperature, °C.
100.0	-12.0*	...
95.0	-14.0*	...
90.0	-15.0*	...
75.0	-14.0*	...
60.0	-11.0*	...
54.5	5.0*	6.0*
50.0	13.4-.2	13.6-.7
37.5	28.4-.2	28.3-.4
25.0	33.4	33.4-.5
12.5	36.2	36.3-.6
5.0	32.4-32.0	32.2-.4
1.25	20.6-.4	20.6-.8

\*Approximate

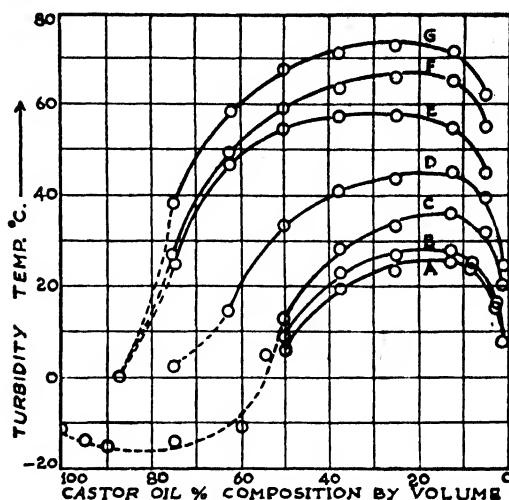


FIG. 1 — MISCIBILITY OF PETROLEUM PRODUCTS  
CURVES A, B AND C REFER TO PETROLEUM ETHERS  
D: KEROSENE; E: SHELL BD<sub>2</sub> LUBRICATING OIL  
F AND G: WHITE OIL I AND II.

In all the cases, the temperatures at which turbidity just disappeared were also noted.

**Results** — Although in most cases the appearance of turbidity was sharp and repeated checks gave fairly concordant values (difference not greater than 0.2°C.), there were instances where the turbidity was either difficult to mark or where no white turbidity appeared, but "liquid droplets" separated on cooling. In such cases, check readings differed by as much as 0.5°C.

Table III gives the experimental results for one binary system. The values for this and other systems are represented graphically in Fig. 1.

### Discussion

The turbidity curves are remarkably similar. In all the cases, mixtures miscible at room temperature are obtained only in high concentrations of castor oil. As the proportion of solvent is increased, the mixtures become miscible only at elevated temperatures. They again become homogeneous at room temperature in very low concentrations of castor oil. The curves show that the 3 kinds of petroleum ethers, normally used in the laboratory, are completely miscible with castor oil at room temperature in equal proportions or in any larger proportion of castor oil. Although curve C only has been studied over the whole range, it will be seen that the other curves follow a course almost parallel to

it over the partial range that has been studied. Castor oil is liable to be mixed either with technical white oils if it is being sold as a hair oil or with kerosene or lubricating oils if it is being sold for lubricating purposes. The curves show that these mineral oils, when mixed with castor oil up to 10-20 per cent of its volume, cannot be visually detected, since such mixtures remain perfectly clear at average room temperature.

The miscibility of castor oil with a petroleum product appears to be related to the Aniline point of the latter. In Fig. 1, the lower-most curve is for petroleum ether having an Aniline point of 53.7°C., the topmost curve is for white oil with an Aniline point of 97.5°C. The 3 petroleum ethers, A, B and C, were found to be almost free from unsaturated components and may be supposed to contain octanes, n-heptane and n-hexane respectively as their major components. Had they been of the highest purity, their Aniline points should have been 71.7°C., 69.7°C. and 68.6°C. respectively. These not only differ considerably from the values obtained for the 3 ethers used, but their order is also reversed. This indicates that even the so-called "chemically pure" petroleum ethers are not only mixtures of straight-chain paraffin isomers, but they

may as well contain cyclo-paraffins and naphthenes. That the miscibility of castor oil depends on the Aniline point, i.e. the paraffinic nature of the solvent, was verified in the case of another lubricating oil with an Aniline point of about 105°C. This oil appeared to be much less miscible than the white oil with an Aniline point of 97.5°C.

#### Summary

Miscibility of castor oil with 3 types of petroleum ethers, kerosene, white oils and lubricating oils has been studied. It has been found that all of them are miscible at average room temperature with castor oil in high as well as in very low concentrations of the oil. The miscibility in the intermediate regions depends on the Aniline point of the petroleum product; the lower the Aniline point, the higher is the miscibility.

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## Chemical Examination of the Seeds of *Cucumis sativus* Linn.

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**C**UCUMIS *sativus* Linn. (Hindi: *Khira*) belongs to the natural order *Cucurbitaceae*. Its seed kernels are edible and are used in confectionery in India. In order to evaluate their nutritive value, a detailed chemical examination of the seed kernels has been undertaken, and the results obtained are recorded in this paper.

A few of the characteristics of the oil have been reported by Hooper<sup>1</sup>. The seed kernels contain about 42.5 per cent oil with the following fatty acid composition:

	%
Acids lower than palmitic	0.63
Palmitic acid	4.14
Stearic acid	16.42
Linoleic acid	40.11
Oleic acid	38.70

The kernels contain about 42 per cent proteins. The ash is rich in phosphates (0.62 per cent  $P_2O_5$ ).

The unsaponifiable fraction of the oil has been crystallized from alcohol in white needles (m.p.  $161^{\circ}$ - $162.5^{\circ}$ C.). It gives a well-defined Libermann-Burchard colour test for sterols. Its acetate melts at  $162^{\circ}$ - $163^{\circ}$ C.

The oil belongs to the semi-drying class. In view of the fatty acid composition, high percentage of protein and appreciable quantity of phosphates, the seed kernels of *Cucumis sativus* should have appreciable food value.

### Experimental

The kernels form 75 per cent of the seeds. The powdered kernels were extracted 4 times with petroleum ether (B.P.  $40^{\circ}$ - $60^{\circ}$ C.). After removing the solvent, 42.50 per cent of a clear, light-yellow, bland oil having the following characteristics was obtained:

		According to Hooper
Sp. gr.	0.9130 ( $40^{\circ}$ C.)	0.9240 ( $15^{\circ}$ C.)
Viscosity at $40^{\circ}$ C.	27.32 centistokes	...
Ref. index at $25^{\circ}$ C	1.4605	...
Acid value	0.22	11.5
Sap. value	193.0	195.2-196.9
Iodine value (Wijs)	114.9	117.7-118.5
Acetyl value	3.1	...
Hegner value	94.86	94.4
Soluble fatty acids calculated as butyric acid, %	0.4	...
Hexabromide value	nil	...
Unsaponifiable matter %	0.91	...
Saturated fatty acids (modified Bertram method)*	20.76%	...

200 gm. of the oil were saponified with alcoholic potash, the alcohol was distilled off and the residual soap dissolved in water. The unsaponifiable matter was removed

TABLE I

FATTY ACIDS	QUANTITY %	NEUTRALIZATION VALUE	IODINE VALUE
Total	...	202.7	116.1
Soluble lead salt	79.49	204.4	138.18
Insoluble lead salt	20.51	201.3	8.1

TABLE II

Weight of esters distilled, 55.0 gm.

FRACTION	TEMPERATURE RANGE $^{\circ}$ C.	PRESSURE, mm.	WT. OF FRACTION gm.
L-1	150-180	6.0	1.48
L-2	180-194	6.0	6.89
L-3	186-190	4.0	9.03
L-4	190-194	4.0	7.41
L-5	194-198	4.0	8.55
L-6	198-210	4.0	8.73
L-7	210-219	4.0	2.47
L-8	Residue	...	9.70
			54.26

with ether and the soap solution decomposed with dilute sulphuric acid. When heated on the water bath, the fatty acids formed a clear oily layer at the top. They were removed with ether and dried in vacuum at  $100^{\circ}$ C.

The acids (150 gm.) were dissolved in 95 per cent alcohol (750 c.c.), the solution boiled and mixed with a boiling solution of lead acetate (110 gm.) in 95 per cent alcohol (750 c.c.), the alcohol in both cases containing 1.5 per cent glacial acetic acid. The insoluble lead salts obtained on cooling overnight at  $15^{\circ}$ C. were crystallized from alcohol containing 1.5 per cent glacial acetic acid and recrystallized from ether at  $0^{\circ}$ C. when a pale-yellow crystalline material was obtained. The solid acids were regenerated from the lead salts. The liquid acids were recovered from the lead salts left over on evaporation of the mixed ether and alcohol mother liquors. The quantities of soluble and insoluble lead salts of fatty acids and some of their characteristics are given in Table I.

TABLE III

FRACTION	CORRECTED WT. gm.	IOD. VALUE	SAP. VALUE	MEAN MOL. WT.	WEIGHT OF ESTERS					
					Methyl caprylate gm.	Methyl caprate gm.	Methyl myristate gm.	Methyl palmitate gm.	Methyl linoleate gm.	Methyl oleate gm.
L-1	1.50	107.09	220.42	254.0	0.17	0.12	...	...	0.67	0.54
L-2	6.98	129.97	195.06	287.6	...	...	0.09	0.04	3.70	3.15
L-3	9.15	138.92	190.46	294.6	...	...	...	...	5.62	3.53
L-4	7.51	140.28	190.37	294.7	...	...	...	...	4.73	2.78
L-5	8.67	142.99	190.26	294.8	...	...	...	...	5.73	2.94
L-6	8.85	140.76	189.78	295.6	...	...	...	...	5.62	3.23
L-7	2.51	120.97	189.77	295.6	...	...	...	...	1.02	1.49
L-8	9.83	91.33	189.36	296.3	...	...	...	...	0.65	9.18
Total	55.00	...	...	...	0.17	0.12	0.09	0.04	27.74	26.84

**Soluble Lead-salt Fatty Acids**

The soluble lead-salt fatty acids, on oxidation with potassium permanganate (modified Bertram method<sup>2</sup>), gave 1.0 per cent saturated acids.

The liquid acids were converted into methyl esters, and a known weight was fractionally distilled under reduced pressure<sup>3</sup>. The results obtained are given in Table II.

The loss in distillation (0.74 gm.) was proportionately divided and added to each fraction, the saponification and iodine values were determined and the amounts of individual esters in each fraction were calculated. The results are given in Table III.

The acids from L-3 and L-5 were oxidized with dilute alkaline potassium permanganate when tetrahydroxy stearic acid (m.p. 172°-173°C.) and dihydroxy stearic acid (m.p. 130°-131°C.) were isolated from both the fractions. Fraction L-8 gave dihydroxy stearic acid by this treatment. The composition of the soluble lead-salt fatty acids is follows :

	%
Acids lower than palmitic	0.67
Palmitic acid	0.06
Linoleic acid	50.44
Oleic acid	48.83

The bromine addition derivatives of the soluble lead-salt fatty acids were prepared according to the method of Eibner and Muggenthalor<sup>5</sup> as described by Jamieson and Boughmann<sup>6</sup>. The results are as follows :

Soluble lead-salt acids taken for analysis	3.1563 gm.
Saturated acids from Bertram's method	0.03 "
Linoleic tetrabromide insoluble in petrol ether, m.p. 113°-114°C.	1.5841 "
Residue (tetrabromide, dibromide and saturated acids)	4.2352 "
Bromine content of the residue	43.33%
Linoleic tetrabromide in the residue	1.8292 gm.
Oleic dibromide in the residue	2.3760 "

Total tetrabromide found	3.4133 gm.
Linoleic acid equivalent to tetrabromide =	1.5930 or 50.47%
Oleic acid equivalent to dibromide =	1.5164 gm. or 48.04%

The mean values obtained for these acids from distillation and bromination results are :

	%
Acids lower than palmitic	0.79
Palmitic acid	0.07
Linoleic acid	50.46
Oleic acid	48.68

**Insoluble Lead-salt Fatty Acids**

The insoluble lead-salt acids, after conversion into methyl esters, were distilled under reduced pressure. The fractions obtained are given in Table IV.

**TABLE IV**

Weight of esters distilled, 22.0 gm.

FRACTION	TEMPERATURE RANGE °C.	PRESSURE mm.	WT. OF FRACTION gm.
S-1	150-175	6.0	5.60
S-2	175-183	6.0	5.65
S-3	176-183	5.0	4.93
S-4	Residue	...	4.86

The loss in distillation (0.96 gm.) was added proportionately to each fraction. The iodine values, saponification values and mean molecular weights of all the fractions were determined. The amounts of various acids in the different fractions were calculated according to the method of Jamieson and Boughmann<sup>6</sup> (TABLE V).

The acids from fraction S-1, on repeated crystallization from alcohol and acetone, gave a product melting from 60° to 67°C., showing it to be a mixture of palmitic acid (m.p. 63°C.) and stearic acid (m.p. 69°C.). The acids from fraction S-4, on crystallization from alcohol, gave stearic acid.

**TABLE V**

FRACTION	CORRECTED WT. gm.	IOD. VALUE	SAP. VALUE	MEAN MOL. WT.	WEIGHT OF ESTERS		
					Methyl palmitate gm.	Methyl stearate gm.	Unsaturated esters gm.
S-1	5.85	2.59	195.18	287.6	2.25	3.48	0.12
S-2	5.91	3.43	193.04	290.6	1.61	4.15	0.15
S-3	5.16	4.04	189.47	295.4	0.39	4.61	0.16
S-4	5.08	8.10	188.88	297.9	0.01	4.76	0.31
Total	22.00	...	...	...	4.26	17.00	0.74

The amount of individual saturated components in the insoluble lead-salt fatty acids are :

Palmitic acid	% 19.96
Stearic acid	80.04

From the above analysis, the total fatty acid constituents of the oil from the seed kernel of *Cucumis sativus* Linn. are :

Acids lower than palmitic	% 0.63
Palmitic acid	4.14
Stearic acid	16.42
Linoleic acid	40.11
Oleic acid	38.70

The composition of the oil, therefore, is :

Glycerides of	Acids lower than palmitic	% 0.63
	Palmitic acid	4.12
	Stearic acid	16.30
	Linoleic acid	39.62
	Oleic acid	38.42
	Unsaponifiable matter	0.91

#### Seed Kernel Cake

The cake obtained from the seed kernels, after complete removal of the petrol-ether soluble fraction, was analysed according to the method described in the *Official and Tenta-*

*tive Methods of Analysis*. The following results were obtained :

Moisture	% 8.13
Proteins ( $N \times 6.25$ )	72.53
Ash	9.7
Crude fibre	1.0
Carbohydrates ( by difference )	8.64

The ash, on analysis, gave 11.17 per cent phosphate calculated as  $P_2O_5$ .

#### Acknowledgement

The authors are thankful to Dr. Siddiqui for his kind interest in the work.

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# Letters to the Editor

## A NOTE ON PULSED EMISSION FROM OXIDE-COATED CATHODE

MECHANISM OF THERMIONIC EMISSION FROM oxide-coated cathode as also that of conduction through oxide coating had been a subject of controversy for a long time. The situation, however, became simpler when Fowler-Wilson<sup>1</sup> theory of semi-conductors based on statistical mechanics was applied to the problem. Electronic energy levels inside a semi-conductor (n-type) are shown in Fig. 1. With the values as shown in the figure, the expression for conductivity  $\sigma$  of the semi-conductor can be shown to be of the form:

$$\sigma = \sigma_0 \exp. (-eQ_{1/2}/kT) \quad (1)$$

where  $e$  is electronic charge,  $k$  is Boltzmann's constant,  $T$  is the temperature of the cathode in  $^{\circ}\text{K}$ ,  $\sigma_0$  is a constant directly proportional to electronic charge and mobility and is very slightly dependent on temperature.  $Q_{1/2}$  is called the activation energy of the

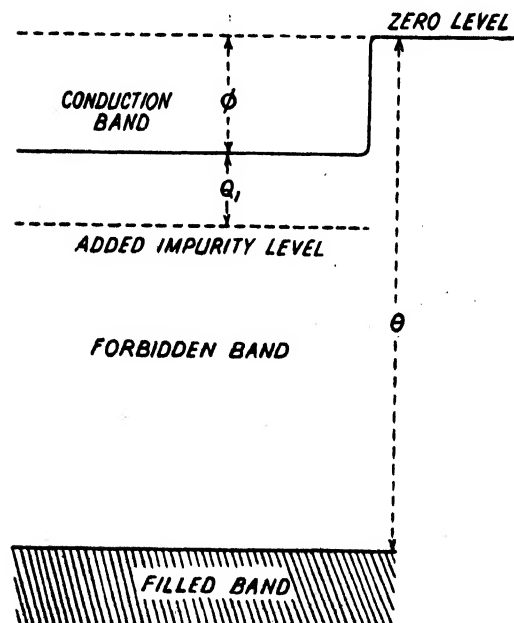


FIG. 1 — ENERGY LEVELS IN AN IMPURITY SEMI-CONDUCTOR (EXTRINSIC N-TYPE).

semi-conductor. The expression for saturated thermionic current  $i_m$  can be written as

$$i_m = \alpha AT^2 \exp. [-e(Q_{1/2} + \phi)/kT] \quad (2)$$

where  $\alpha$  is a constant slightly dependent on temperature,  $A$  is the usual constant of Dushmann's equation and is independent of temperature. This approach to the problem helped to explain many peculiarities observed in an oxide-coated cathode and could predict emission of the correct order of magnitude. In recent years, however, peculiar effects were observed in an oxide-coated cathode operated with pulsed voltage of microsecond duration on the anode. It was found that surprisingly large amount of peak current could be drawn from a cathode which, under condition of d.c. operation, gives a much lower magnitude of current. This effect aroused a lot of speculation among the workers and it has led to the suspicion that Fowler-Wilson theory of semi-conductor is perhaps not the correct approach to the problem, at least under pulsed operation. For example, Blewett<sup>2</sup> has suggested that the large emission observed is probably connected with some non-equilibrium condition inside the cathode and, therefore, statistical-mechanical approach, which is valid only under condition of equilibrium, may not be applicable to such a case. He and Hahn have, therefore, advocated a new approach based on mass motion. An interesting suggestion due to Coomes<sup>3</sup>, however, does not require such a radical revision of idea. He has suggested that electrons which are thermally excited to conduction level during the period between the successive pulses are swept away by the pulse, thus causing a momentary flow of a large amount of current. This suggests that expression for conductivity of the oxide layer as also of thermionic emission current should be given by expressions of the type (1) and (2). Experimental data available to date are not, however, sufficient for an outright decision in favour of one or the other suggestion. In a recent communication Danforth and Goldwater<sup>4</sup> have given some interesting experimental data for pulses of microsecond duration. They found that  $\log 1/R$  plotted

against  $1/T$  gives a straight line whose slope, within experimental accuracy, lies near about the value of  $Q_1$  obtained by other workers under condition of d.c. operation by using relation of the type (1) ( $R$  being the resistance of the cathode). In their experiment they used a probe embedded inside the oxide coating to determine  $R$ . An interesting method of measurement which avoids complications due to the embedded probe was used by Loosjes and Vink<sup>5</sup> to determine the characteristics of an oxide-coated cathode for pulses of duration  $10^{-4}$  sec. They found that surprisingly large amount of potential drop exists across the oxide layer under such condition of operation. They have not, however, examined if the data obtained by them obey relations of the type (1) and (2). This has been done and is being presented in this note. It is found that there is strong evidence that the Fowler-Wilson theory of semi-conductor might be applicable in pulsed operation and, therefore, suggestion offered by Coomes deserves serious consideration.

Current voltage characteristics ( $i-v$ ) of the cathode given by Loosjes and Vink show the following 3 distinct regions:

1. A very small linear region at the origin.
2. A curved region beyond (1).
3. A nearly linear region for high value of  $V$ .

Curve (1) in Fig. 2 has been drawn for the first region. Conductivities were calculated by drawing a tangent at the origin of the characteristic. It is seen that the curve is essentially linear with a slope of 1.05 volts. Curve (2) has been drawn for the second region mentioned above. Conductivities were calculated for  $V = 20$  volts. Here also  $\log \sigma - 1/T$  is found to be linear. Slope of the curve is about 1.4 volts. Curve (3) has been drawn for the third region mentioned above. The curve is linear with a slope of 1.1 volts. It is seen that the slope of  $\log \sigma - 1/T$  curve for region (2) differs from the other two values. Conductivities calculated for this region from the values of  $i$  and  $v$  at point should, however, be interpreted carefully. Characteristics is curved in this region, which shows that a non-ohmic element in the cathode might be playing an important rôle in this region. It is well known that non-ohmic contacts give rise to a potential barrier having the property of rectification. Presence of a fairly thick barrier of this type

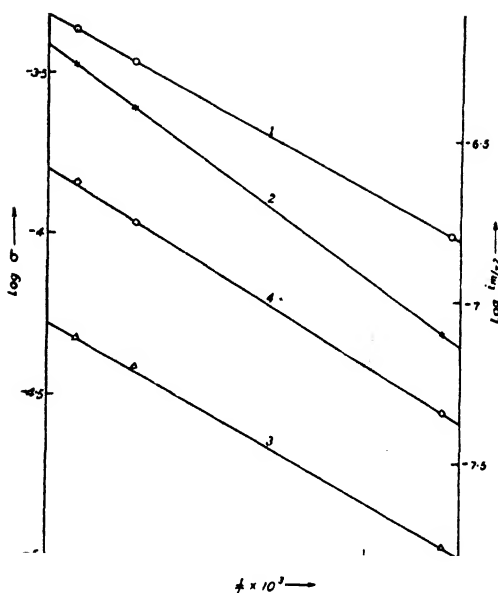


FIG. 2 — FIGURE SHOWING CONDUCTIVITY AS FUNCTION OF TEMPERATURES (CURVES 1, 2, 3 AND RICHARDSON PLOT OF EMISSION CURRENT (CURVE 4)).

in the core/oxide interface of an oxide-coated cathode has been definitely established by Fineman and Eisenstein<sup>6</sup>, Wright<sup>7</sup> and Coomes<sup>3</sup>. Resistance of this barrier may be high compared to that of the semi-conductor. The linear relationship obtained for this region in curve (2) also indicates that the combined effect of a potential barrier at the core/oxide interface and the semi-conducting oxide layer is to give rise to a resistance having a higher activation energy. This is also expected from theoretical expression given by Mott<sup>8</sup> for the current flowing through a potential barrier of the type mentioned above. Therefore, leaving the value for the curved region out of consideration we can take 1.1 volts as the activation energy for the semi-conducting oxide layer. This does not agree well with the value of .825 volt given by Nishibori and Kawamura<sup>9</sup> for d.c. operation, but agrees well with the value of  $1.2 \pm 1$  volt found by Mahlman<sup>10</sup>. With 1.1 volts as the activation energy of the semi-conductors, and hence  $Q_1 = 2.2$  volts, the impurity level must be about 2.2 volts below the bottom of the conduction band and, therefore, the energy separation between the top of the filled band and the bottom of the conduction band is at least 2.2 volts. Calculation of Wright<sup>11</sup> on the basis of the treatment of Mott and Gurney<sup>12</sup> for alkali

halides gives for BaO a value of 0 (FIG. 1) the energy separation between the zero level and the topmost level of the filled band as 2.6 volts. This shows that  $\phi$  should have a value of .4 or less. According to Wright, the maximum value that  $\phi$  can have is .6 for BaO, but it may be zero or even negative if the bottom of the conduction band lies above the zero level. Supposing, therefore, that the value of .4 is quite admissible, one finds that the maximum value of thermionic work function is

$$Q_{1/2} + \phi = 1.5 \text{ volts}$$

This value can be roughly checked with the help of the data given by Loosjes and Vink. From the values of plate voltage and thermionic current given, it is possible to determine  $i_m$  the maximum possible emission for the cathode at a particular temperature without Schottky effect. This is done by simply plotting  $\log i$  against  $\sqrt{\text{plate voltage}}$  and then finding the intercept on the  $\log i$  axis obtained by producing the linear portion of the curve. The following data were thus obtained:

Temp.	$\log i_m$
976°K	-1.35
1078°K	-.70
1100°K	-.56

Curve was drawn with  $\log i_m/T^2$  as ordinate and  $1/T$  as the abscissa and curve (4) in Fig. 2 was obtained. This is a straight line as required by equation (2). From the slope of the line the value of thermionic work functions is found to be 1.19 volts. This is within the limit set by previous consideration. This value can be compared with the values of 1.15 volts obtained with d.c. operating voltage.

It is also possible to determine the value of " $\alpha A$ " from the intercept made on the

$\log i_m/T^2$  axis by curve (4). The value thus obtained roughly is .08. This is much larger than the usual value of .0013-.000013 found for oxide-coated cathode under d.c. operating conditions, but is only slightly greater than the value of .03 found by Mahlman<sup>10</sup>. It is thus seen that the larger emission obtained under pulsed condition is due to a large increase in the factor " $\alpha A$ " and not at all due to any lowering of work function. Indeed, there are evidences which show that for the same cathode the work function is probably larger under condition of pulsed operation.

It is a pleasure to acknowledge with thanks the encouragement received from Prof. M. N. Saha and helpful suggestions and guidance received from Prof. S. K. Mitra in course of this work.

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August 25, 1949

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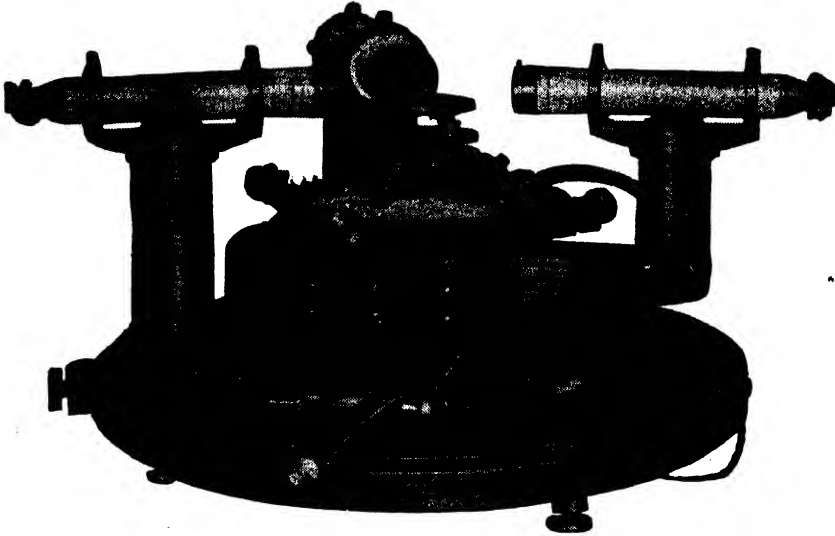
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# Isoflavones from the Fresh Soya Bean Germ & the Synthesis of 6-Methyl-Formononetin & 6-Methyl-Daidzein

PRITHVI RAJ BHANDARI, JOGENDRA LAL BOSE & SALIMUZZAMAN SIDDIQUI

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AS many as eight isoflavones, representing about half the total number of isoflavones so far isolated from plant materials, have been found to occur in soya beans (*Soja hispida*). In the course of his investigations on soya bean meal, Walz<sup>1</sup> isolated four isoflavone glucosides, the aglucones of two of which are genistein and daidzein. Although no definite constitution was arrived at for the other two aglucones, their isoflavone nature was clearly established. Later, Okano and Beppu<sup>2</sup> isolated from the same source four new isoflavones, two of which, namely tatoin and methyl-genistein, were obtained in the free state, although the method of isolation employed by them leaves doubt as to their occurrence in the free state in the plant body. The other two, namely methyl-isogenistein and isogenistein were isolated as glucosides. The constitution forwarded for methyl-genistein, namely that of 5, 7, 4'-trihydroxy-8-methylisoflavone has since been confirmed by a synthesis of the latter by Shriner and Hull<sup>3</sup>. For tatoin, a constitution of 5, 4'-dihydroxy-8-methylisoflavone has been advanced, but has yet to be confirmed by synthesis.

The isolation of biochanins A, B and C from the fresh germ of germinated *chana*<sup>4</sup> and *Kabuli chana*<sup>5</sup> (*Cicer arietinum* Linn.; ordinary and white varieties), of which the constitution of biochanin A was established as 5, 7-dihydroxy-4'-methoxy-isoflavone<sup>6</sup>, led to the present investigations on the constituents of the fresh germ of various Indian

pulses. In this connection it was considered of special interest to extend these investigations to the fresh germ of soya bean<sup>7</sup>, which occupies in the Far Eastern countries a rôle in dietetics similar to that of *chana* in India.

A small variety of soya beans (Penygphe type), grown in India and obtained from the Superintendent, Agricultural Research Station, Maruter (West Godavari), Madras, were used in the present investigations. The fresh germs of germinated soya beans were worked up in the same manner as the fresh germ of *chana* through dialysis with alcohol, and three crystalline substances were isolated, one of which was found to be identical with biochanin C<sup>7</sup>. Of the other two substances, the sparingly soluble one (substance B) was obtained in colourless prismatic rods, m.p., 322°-323°C. (decomp.), in a yield of 0.001 per cent on the weight of the dry grain. It appears to be of the nature of an isoflavone and further work on its constitution is in progress. The more soluble product, substance A, was obtained in colourless prismatic rods, m.p., 316°-317°C. (yield, 0.004 per cent on the weight of the dry grain), which analysed for the molecular formula  $C_{16}H_{12}O_4$ . An alcoholic solution of the substance gave a brownish colouration with ferric chloride. Its hydrolysis with 15 per cent aqueous-alcoholic potassium hydroxide solution yielded formic acid and p-hydroxyphenylacetic acid which did not depress the m.p. of a synthetic sample of the acid. Substance A further yielded a diacetyl derivative, m.p., 185°C., and appears to be

identical with tatoon, m.p., 318°C., the diacetyl derivative of which also melts at 185°C.<sup>2</sup>

As an extension of the work referred to above, it was considered of interest to carry out some related synthetic studies in the isoflavone series. The synthesis of isoflavones by condensation of phenyl benzyl ketones with absolute ethyl formate in presence of sodium dust was first employed by Späth and Lederer<sup>8</sup> in the synthesis of daidzein and pseudo-baptigenin, by heating the reaction mixture in a sealed tube at 100°C. Wessely, Kornfeld and Lechner<sup>9</sup> have also used this method in the synthesis of formononetin and daidzein. Venkataraman and co-workers<sup>10</sup> have considerably improved upon the earlier method by allowing the reaction to take place at 0°C. for a longer period. The *O*-benzyl ethers of the ketones were used in this method and thus two more steps were involved, namely benzylation of the ketones and debenylation of the isoflavones formed. Shriner and Hull in their synthesis of methyl-genistein<sup>3</sup> and 5, 7-dihydroxy-4'-methoxy-isoflavone (biochanin A)<sup>6</sup> modified Venkataraman's method by eliminating the steps of benzylation and debenylation, and this modification was adopted in the present work.

Cresorcinol and homoanisonitrile were subjected to the Hoesch reaction and the intermediate ketimine hydrochloride (I) was hydrolysed to 2, 4-dihydroxy-5-methylphenyl 4-methoxybenzyl ketone (II), colourless prismatic rods, m.p., 139°-140°C. That ketone (II) is formed in the reaction and not 2, 6-dihydroxy-3-methylphenyl 4-methoxybenzyl ketone (which probably would have led to a synthesis of tatoon) is supported by earlier work in the field<sup>11</sup> which has established that, in the formation of dihydroxyphenyl benzyl ketones by the Hoesch reaction, the —CO— group is in the *o-p* position with respect to the phenolic groups, which may be either free or substituted. Ketone (II) which was characterized through a crystalline oxime, m.p., 165°C. (decomp.), was allowed to react with absolute ethyl formate in presence of sodium dust at 0°C. The intermediate compound (III) thus formed gave on acidification the isoflavone (IV), namely 6-methyl-formononetin, in small colourless prismatic rods, m.p., 260°C. On demethylation with hydroiodic acid it yielded 6-methyl-daidzein (V) in colourless needles, m.p., 329°-330°C. (decomp.), which gave a diacetyl derivative in colourless prismatic rods, m.p., 200°C.

The synthetic substance thus proved to be an isomer of tatoon.

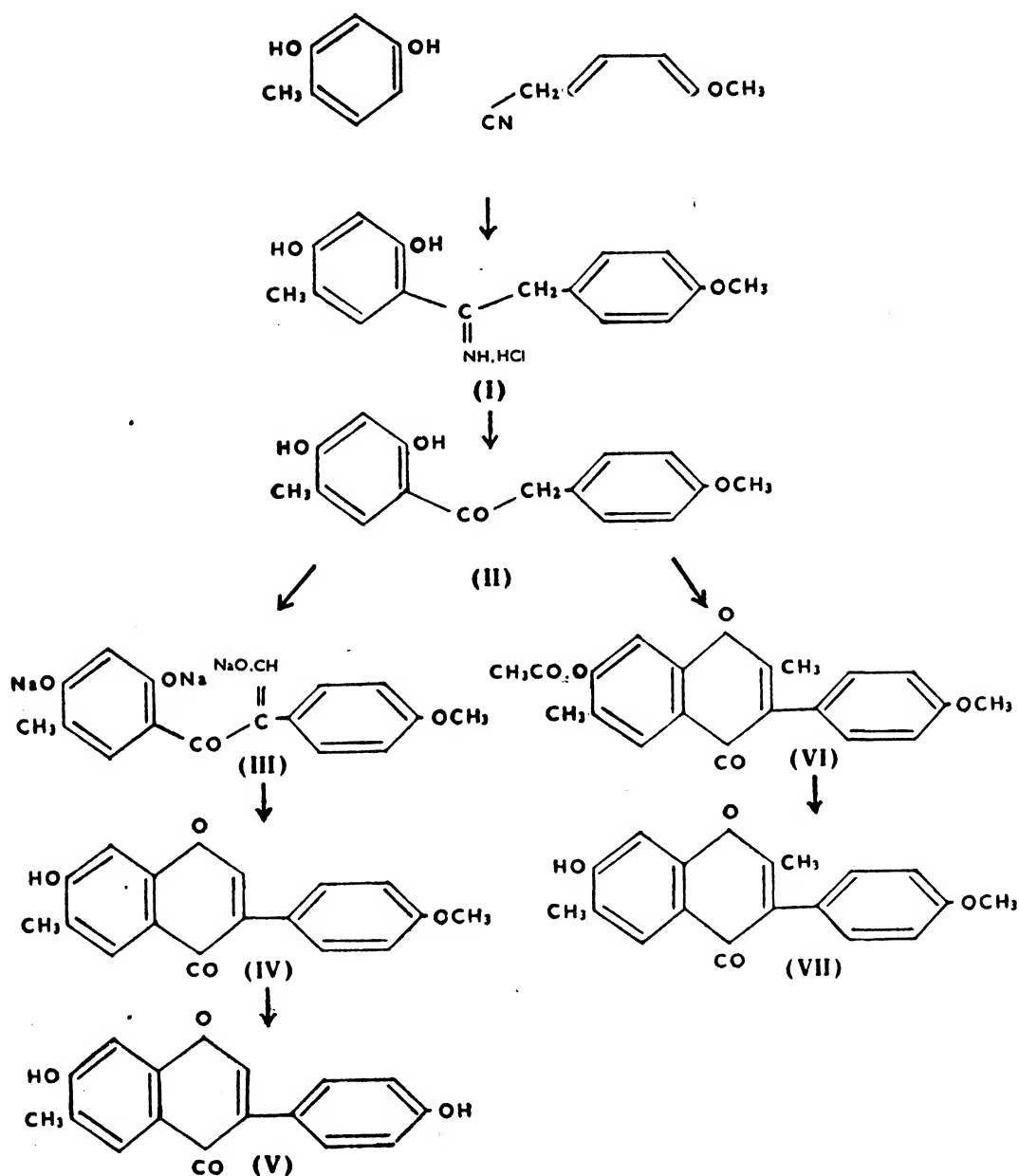
Vigorous acetylation of ketone (II) yielded 7-acetoxy-4'-methoxy-2,6-dimethylisoflavone (VI) in clusters of colourless needles, m.p., 197°C. Deacetylation of (VI), with concentrated sulphuric acid at 0°C., gave 7-hydroxy-4'-methoxy-2, 6-dimethylisoflavone (VII) in small colourless needles, m.p., 257°-258°C.

Cresorcinol was prepared from *p*-toluidine, through *o*-nitro-*p*-toluidine, *o*-nitro-*p*-cresol and *o*-amino-*p*-cresol. Homoanisonitrile was obtained from phenyl-acetonitrile through *p*-nitro, *p*-amino and *p*-hydroxy derivatives.

### Experimental

**Isolation & Characterization of the Crystalline Products** — 5 kg. of soya beans (in batches of 0.5 kg. each) were thoroughly washed and soaked in water for 24 hr. and then allowed to germinate in enamelled trays in 1 in. thick layers at the room temperature (18°-25°C.). The grains were kept covered with moist filter paper and were raked carefully at regular intervals for proper aeration. After 96-100 hr. of germination, the germs attained an average length of 1 in. and were separated by handpicking. During picking, the germs were kept moist by keeping them on wet filter paper and a total of 1250 gm. of the fresh germ was obtained.

The technique of isolation employed was similar to that used for the *chana* germ. The fresh whole germ was dialysed four times with alcohol (5 litres) at the room temperature. The first and the second dialysates were removed after intervals of 5 hr. each and the other two at intervals of 24 hr. each. The combined dialysates, on keeping, deposited some colourless crystalline matter which was identified as biochanin C. On removal of the solvent *in vacuo*, the concentrate deposited a solid on keeping for a few days. This was filtered and mixed with the concentrate of the ether extractive of the filtrate and the combined solid was repeatedly extracted with petroleum ether to remove fatty matter. The petroleum ether-insoluble part (0.4 gm.), on fractionation through alcohol, gave 0.05 gm. of substance B, crystallizing in colourless prismatic rods, m.p., 322°-323°C. (decomp.), in the less soluble fraction. The more soluble fraction yielded 0.2 gm. of substance A in colourless prismatic rods, m.p., 316°-317°C. A mixture of equal quantities of substances A and B melted at 302°-305°C.



The analytical data of substance A is given below:

Found after drying to constant weight at  $100^\circ\text{C}$ . *in vacuo* over phosphorus pentoxide, C, 71.3; H, 4.2;  $\text{C}_{16}\text{H}_{12}\text{O}_4$  requires C, 71.6; H, 4.5 per cent.

The ether-insoluble portion of the concentrated dialysate left over after the removal of the crude mixture of substances A and B and after the separation of the products insoluble in 50 per cent alcohol yielded a

product which, on repeated crystallizations from dilute alcohol, yielded colourless prisms, m.p.,  $310^\circ\text{C}$ . (with decomposition and darkening earlier), and was found to be identical with biochanin C. The yield of the crude substance was 0.1 per cent on the weight of the dry grain.

*Acetyl Derivative of Substance A* — A mixture of substance A (0.05 gm.), freshly fused sodium acetate (0.5 gm.) and acetic anhydride (1 c.c.) was refluxed on the



boiling water bath for 2 hr. After cooling, the reaction mixture was poured into water and left overnight when the crude acetyl derivative separated out. For complete separation, the solution was neutralized with sodium bicarbonate and extracted with ether. The residue left over after the removal of ether was repeatedly crystallized from dilute alcohol when the pure acetyl derivative was finally obtained in colourless needles, m.p.,  $185^{\circ}\text{C}$ . (yield, 0.04 gm.).

Found after drying to constant weight at  $100^{\circ}\text{C}$ . *in vacuo* over phosphorus pentoxide, C, 67.8; H, 4.3;  $\text{C}_{20}\text{H}_{16}\text{O}_6$  requires C, 68.2; H, 4.5 per cent.

**Hydrolysis of Substance A: Isolation of *p*-hydroxyphenylacetic Acid & Detection of Formic Acid**—Substance A (0.1 gm.) was dissolved in a 15 per cent solution of aqueous-alcoholic potassium hydroxide (10 c.c.) and was refluxed on the water bath for 5 hr. After cooling, the reaction mixture was diluted with water (20 c.c.) and was acidified with dilute phosphoric acid under cooling, and extracted 5 times with petroleum ether (75 c.c.). On removal of petroleum ether, glassy prismatic crystals of an acid were obtained. It was purified by crystallizing from benzene-petroleum ether mixture and melted at  $147^{\circ}\text{--}148^{\circ}\text{C}$ . There was no depression in its melting point on admixture with an authentic sample of *p*-hydroxyphenylacetic acid, m.p.,  $148^{\circ}\text{C}$ .

The aqueous layer was subsequently steam-distilled. The distillate was acidic in nature and was neutralized with sodium carbonate, concentrated to a small volume on the water bath and was acidified with dilute hydrochloric acid (1:1). It gave the following test for formic acid:

A 2 c.c. portion of the acidic solution was reduced with magnesium and then 2 c.c. of fresh milk added to it followed immediately by the addition of 0.5 c.c. of concentrated sulphuric acid along the sides of the test-tube. A deep-violet ring was obtained at the junction of the two liquids. Parallel experiments with distilled water and very dilute formic acid were made for comparison.

**2, 4-Dihydroxy-5-methylphenyl 4-methoxybenzyl Ketone**—A mixture of cresorcinol (1.1 gm.), homoanisonitrile (1.1 gm.), freshly fused zinc chloride (0.5 gm.) and absolute ether (20 c.c.) in a 50 c.c. flask was saturated with dry hydrochloric acid gas for 4 hr. at  $0^{\circ}\text{C}$ ., and kept in a refrigerator for 2 days. The supernatant ethereal solution was decanted off and the

yellow residue consisting mainly of the ketimine hydrochloride was washed twice with absolute ether and was hydrolysed by refluxing with 1 per cent sulphuric acid (110 c.c.) for 1 hr. The colour of the solution changed during the hydrolysis from yellow to green, but on cooling the green colour disappeared and the crude ketone separated out as a nearly colourless semi-solid mass which solidified on keeping in a refrigerator. It was filtered and the aqueous filtrate was extracted with ether. The residue left over after the removal of ether was taken up with the main fraction of the crude ketone and repeatedly crystallized from dilute methanol when the pure ketone was finally obtained in colourless prismatic rods, m.p.,  $139^{\circ}\text{--}140^{\circ}\text{C}$ . A further quantity of the ketone was obtained by hydrolysis of the ketimine hydrochloride which separated out in the decanted ether and ethereal washings on the addition of more absolute ether and keeping. Total yield of the ketone was 0.9 gm.

Found after drying to constant weight at  $58^{\circ}\text{C}$ . *in vacuo* over phosphorus pentoxide, C, 70.1; H, 5.8;  $\text{OCH}_3$ , 11.4;  $\text{C}_{18}\text{H}_{16}\text{O}_4$  requires C, 70.6; H, 5.8;  $\text{OCH}_3$  (for one methoxy), 10.8 per cent.

**Oximation of 2, 4-dihydroxy-5-methylphenyl 4-methoxybenzyl Ketone**—The ketone (0.1 gm.) was dissolved in pure methanol (10 c.c.) and finely powdered hydroxylamine hydrochloride (0.6 gm.) was added to it followed immediately by freshly fused sodium acetate (1 gm.). The mixture was refluxed on the water bath for 5 hr. at  $70^{\circ}\text{--}80^{\circ}\text{C}$ ., after which it was cooled and diluted with ice-cold water (50 c.c.) when the crude oxime separated out as a colourless solid. On repeated crystallizations from 1:1 aqueous methanol (charcoal), the pure oxime was obtained in colourless needles, m.p.,  $165^{\circ}\text{--}166^{\circ}\text{C}$ . (decomp.) (yield, 0.07 gm.).

Found after drying to constant weight at  $58^{\circ}\text{C}$ . *in vacuo* over phosphorus pentoxide, C, 66.9; H, 5.9; N, 4.9;  $\text{C}_{18}\text{H}_{17}\text{O}_4\text{N}$  requires C, 66.6; H, 6.1; N, 4.7 per cent.

**6-Methyl-formononetin (7-hydroxy-4'-methoxy-6-methylisoflavone)**—To sodium dust (0.3 gm.) covered with absolute ethyl formate (2 c.c.) and kept at  $0^{\circ}\text{C}$ ., was added 2, 4-dihydroxy-5-methylphenyl 4-methoxybenzyl ketone (0.58 gm.) dissolved in absolute ethyl formate (12 c.c.) during the course of 3 hr. The contents of the flask were well stirred during the addition and for another 4 hr. after the addition

was complete, and allowed to remain in a refrigerator for 2 days. Thereafter it was acidified with ice-cold hydrochloric acid (6N) and small pieces of ice were added at intervals during acidification. On removal of ethyl formate under vacuum, a pasty mass separated out which solidified on keeping for 1 day in the refrigerator. The crude isoflavone thus obtained was crystallized from alcohol repeatedly (charcoal), when the pure substance was obtained in small colourless prismatic rods, m.p., 260°C. (yield, 0.12 gm.).

Found after drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, C, 71.9; H, 4.8;  $C_{17}H_{14}O_4$  requires C, 72.3; H, 5.0 per cent.

**6-Methyl-daidzein (7, 4'-dihydroxy-6-methylisoflavone)** — 6-Methyl-formononetin (0.07 gm.) was dissolved in pure phenol (0.2 gm.) and refluxed with hydriodic acid (8 c.c., d. 1.7) for 2 hr. at 130°-140°C. After cooling, the reaction mixture was poured into water (20 c.c.) containing sodium bisulphite (1 gm.) and left overnight. The crude product thus obtained was repeatedly crystallized from alcohol when 6-methyl-daidzein was finally obtained in colourless needles which begin to darken from 310°C. onwards and melt with decomposition at 329°-330°C. (yield, 0.04 gm.). The isoflavone gave a brownish colouration with ferric chloride in alcoholic solution.

Found after drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, C, 71.3; H, 4.4;  $C_{16}H_{12}O_4$  requires C, 71.6; H, 4.5 per cent.

**7, 4'-Diacetoxy-6-methylisoflavone** — A mixture of 6-methyl-daidzein (0.03 gm.) and freshly fused sodium acetate (0.1 gm.) was refluxed with acetic anhydride (1 c.c.) at 130°-140°C. for 2 hr. After cooling and addition of water (30 c.c.), the reaction mixture was left overnight, after which it was neutralized with sodium bicarbonate and extracted with ether. On removal of ether and repeated crystallization from alcohol, the acetyl derivative of the isoflavone was finally obtained in colourless prismatic rods, m.p., 200°C. (yield, 0.03 gm.).

Found after drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, C, 67.8; H, 4.3;  $C_{20}H_{16}O_6$  requires C, 68.1; H, 4.5 per cent.

**7-Acetoxy-4'-methoxy-2, 6-dimethylisoflavone** — A mixture of 2, 4-dihydroxy-5-methylphenyl 4-methoxybenzyl ketone (0.1 gm.), freshly fused sodium acetate (2 gm.)

and acetic anhydride (15 c.c.) was refluxed at 170°-180°C. for 30 hr. After cooling and the addition of water (100 c.c.), the reaction mixture was left overnight, when a part of the crude isoflavone separated out and was collected. A second fraction of the isoflavone was obtained when the filtrate was neutralized with sodium carbonate and extracted with ether. On crystallization of the total crude product from dilute alcohol (charcoal) the pure 2-methylisoflavone was obtained in clusters of colourless needles.

Found after drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, C, 71.4; H, 5.6;  $C_{20}H_{18}O_5$  requires C, 71.0; H, 5.3 per cent.

**7-Hydroxy-4'-methoxy-2, 6-dimethylisoflavone** — 7-Acetoxy-4'-methoxy-2, 6-dimethylisoflavone (0.04 gm.) was taken in a small flask, cooled in a freezing mixture and was treated with ice-cold concentrated sulphuric acid (0.25 c.c.) drop by drop. The reaction mixture was stirred with a glass rod till a complete solution was effected. After the solution was allowed to attain the room temperature (25°C.) in the course of c. 10 min., crushed ice was added to it when the de-acetylated isoflavone separated out. It crystallized from alcohol in clusters of small colourless needles, m.p., 257°-258°C. (yield, 0.025 gm.).

Found after drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, C, 72.5; H, 5.6;  $OCH_3$ , 10.1;  $C_{18}H_{16}O_4$  requires C, 72.2; H, 5.4;  $OCH_3$  (for one methoxy), 10.5 per cent.

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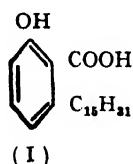
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# Subsidiary Constituents of Cashew Nut Shells—Part I

OM PRAKASH BAHL, RAM PRAKASH RASTOGI,  
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THE resinous pericarp juice of the cashew nut was first investigated in 1847 by Staedeler<sup>1</sup> who reported the isolation from it of an acid, anacardic acid,  $C_{44}H_{32}O_7$ , in a yield of 90 per cent, and a non-acidic, vesicant principle, cardol, to the extent of nearly 10 per cent. Later, in 1887, Ruhemann and Skinner<sup>2</sup> purified anacardic acid, which they considered to be a hydroxy carboxylic acid, and assigned to it its present molecular formula,  $C_{22}H_{32}O_5$ . More recently, as a result of studies in the constitution of anacardic acid, Smit<sup>3</sup> arrived at the conclusion that it is a salicylic acid derivative with a straight, unsaturated  $C_{15}$  side chain. On the basis of subsequent investigations on its reduction, decarboxylation and oxidation, Pillay<sup>4</sup> proposed the structure of *O*-pentadecyl-salicylic acid (I) for tetrahydro-anacardic acid. This structure found support through synthetic studies of Gokhale, Patel and Shah<sup>5</sup>, and Baker and Hack<sup>6</sup>.



Backer and Hack (loc. cit.) also studied the vesicant principle of cashew nut shell liquid, cardol,  $C_{31}H_{32}O_2$ , and considered it to be allied to bhillawanol and urushiol, as it gave a diacetyl derivative and a tetrahydro derivative, which, on oxidation, yielded palmitic acid.

From this brief review it will be seen that although the chemistry of cashew shell liquid has been studied by numerous workers over a long period extending back to 1847, no attention has been so far given to the isolation and study of any subsidiary constituents which may be present in it. It was, therefore, considered of interest to make a systematic study of this aspect of its chemistry. As a result of the present

investigations, the following constituents have been isolated from the alcoholic extract of shells of a one-year old sample of mature cashew nuts:

(1) A crystalline glucoside, kajin,  $C_{18}H_{20}O_9 \cdot 1\frac{1}{2}H_2O$ ; m.p.,  $174^\circ C.$ , decomp.; yield, 0.12 per cent on the weight of the shells.

(2) Gallic acid (present as potassium salt); m.p.,  $241^\circ C.$ , decomp.; yield, 0.06 per cent on the weight of the shells.

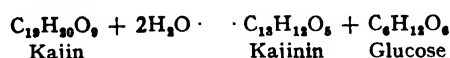
(3) A petroleum ether-insoluble micro-crystalline product, kajidin,  $C_{14}H_{14}O_8 \cdot H_2O$ ; m.p.,  $185^\circ C.$ , decomp.; yield, 0.56 per cent on the weight of the shells.

In place of kajidin, a micro-crystalline substance (m.p.,  $196^\circ C.$ , decomp.) which has been provisionally named as anacardin, was obtained from the shells of a nearly ten-year old sample of cashew nuts.

The isolation of these products was based on fractionation of the alcoholic extract through single and mixed solvents without the use of any chemical reagents. The concentrate obtained from the alcoholic percolates of the crushed shells, on removing the major portion of the solvent *in vacuo*, gave on keeping in the cold a yellowish-brown deposit of crude kajidin. From the supernatant viscid liquid, the glucoside, kajin, and gallic acid were obtained by partitioning between petroleum ether and 80 per cent alcohol according to the procedure described in the experimental.

The glucoside kajin, which crystallizes from dilute alcohol in silky needles, is very sparingly soluble in water, gives violet colouration with ferric chloride and readily dissolves in dilute alkali with a yellow colouration. The substance is recovered unchanged on acidification of the solution. It yields an acetyl derivative,  $C_{27}H_{30}O_{12}$ , with intramolecular elimination of one molecule of water. The sugar component of kajin was identified as glucose through the mixed melting point determination of the osazone with an authentic sample of glucosazone. Attempts to isolate the aglucone in a crystal-

line form through hydrolysis with mineral acids under varying conditions of acid concentration and duration of heating were not successful. Hydrolysis of kajin with baryta solution, however, led to the isolation of a crystalline aglucone, the analytical data of which indicate its formation with the addition of one molecule of water. The course of the hydrolysis can be represented as follows:



Unlike kajin, the aglucone, provisionally named as kajinin, dissolves in sodium bicarbonate solution with effervescence. It is unsaturated to bromine, does not contain any methoxy group, and gives a reddish-brown colouration with ferric chloride. It also yields acetyl and methyl derivatives, neither of which gives any colouration with ferric chloride.

Gallic acid was identified through parallel and mixed melting point determinations of the acid and its derivatives with authentic samples of gallic acid and its corresponding derivatives. Gallic acid, in the form of its metallic salts or its derivatives, is known to occur widely in nature. From among the plants of *Anacardiaceae* family, it has been isolated from the seeds and blossoms of *Mangifera indica*<sup>7</sup>, *Pistacia terebinthus*<sup>8</sup>, *Pistacia lentisus* L.<sup>9</sup>, *Rhus rhodanthema*<sup>10</sup>, and *Rhus coriaria* L.<sup>11</sup>. There is no mention in literature, however, regarding its presence in any of the lacquer-yielding plants.

Kajidin which forms a cream-coloured, micro-crystalline powder, m.p., 185°C., decomp., is soluble in alcohol, and insoluble in ether, benzene or water, is optically inactive, gives an inky-blue colouration with ferric chloride and dissolves in sodium bicarbonate solution with effervescence. It reduces Fehling solution and yields an amorphous tetra-acetyl derivative which does not give any colouration with ferric chloride. On hydrolysis with baryta, kajidin gave gallic acid in a yield of 50 per cent, which would indicate the possibility of its being impure digallic acid. It absorbed hydrogen equivalent to one double bond on catalytic reduction with platinum black, but this may be due to the presence of highly unsaturated impurities.

The colour reactions and solubilities of anacardin were similar to those of kajidin, but the C and H values were found to differ widely for the two compounds. Moreover,

hydrogenation of anacardin gave tetrahydro-anacardic acid, m.p., 88°C. (yield, 25 per cent). On hydrolysis with baryta solution, anacardin gave only 8 per cent gallic acid, and no reducing sugar could be detected among the products of its hydrolysis. It would thus appear that anacardin, which differs from anacardic acid in being completely insoluble in ether, may be a loose molecular complex of anacardic and gallic acids. Further work to elucidate the exact nature of these compounds is in progress.

## Experimental

*Isolation* — 4700 gm. of cashew shells from a fresh decortication of the nuts were repeatedly percolated with alcohol at the ordinary temperature, and the combined extracts were concentrated *in vacuo* below 50°C. The dark-brown, viscous concentrate was kept in the cold for a few days, and the supernatant liquid was decanted off from the semi-solid deposit, which was repeatedly washed with small quantities of petroleum ether and ether till it assumed the form of an amorphous, cream-coloured, granular powder (crude kajidin; yield, 28 gm.). The combined ether and petroleum ether washings were freed of the solvent and the darkish-brown, viscid residue was taken together with the alcoholic decantate (total liquid, 960 gm.) and partitioned between 6.5 litres of petroleum ether and 80 per cent alcohol. The combined brownish-red alcoholic solution (6 l.) was concentrated on a water bath till turbid and left overnight at room temperature. The colourless clusters of rods and needles of kajin, which separated out along with a heavy, dark-brown viscous liquid, were filtered under suction. The viscous deposit was triturated with petroleum ether and also filtered under suction, when most of the matrix went in the petroleum ether solution leaving a colourless crystallizate (crude kajin; yield, 5.5 gm.) which was repeatedly washed with petroleum ether. The petroleum ether washings were added on to the initial alcoholic filtrate which was worked up for the phenolic acid as described later.

*Kajin*,  $\text{C}_{18}\text{H}_{30}\text{O}_9 \cdot 1\frac{1}{2}\text{H}_2\text{O}$  — Crude kajin, as obtained by the procedure described above, m.p., 164°C., decomp., was repeatedly crystallized from ethyl acetate and then from 40 per cent alcohol, when it finally gave pure kajin in the form of colourless, silky needles which shrink at 172°C., and melt with decomposition at 174°C. On drying to constant

weight *in vacuo* over phosphorus pentoxide at 61°C., it lost 6.7 per cent in weight and melted with decomposition at 184°C. after shrinking at 175°C. ( $C_{19}H_{20}O_9 \cdot 1\frac{1}{2}H_2O$  requires loss in weight on drying 6.9 per cent). The dried substance showed  $(\alpha)_D^{25} = -69.0^\circ$  in 1 per cent alcoholic solution.

Kajin is readily soluble in methanol, alcohol and acetone, fairly so in ethyl acetate in the hot, and insoluble in chloroform, ether, petroleum ether and water. It is insoluble in sodium bicarbonate solution, but dissolves in ammonia or dilute alkali with a yellow colouration, and is precipitated unchanged on acidification of the solution. With ferric chloride in alcoholic solution, kajin gives a violet colouration which changes to brown. It dissolves in concentrated sulphuric acid with a yellow colouration, and in concentrated nitric acid with a greenish-blue colouration. Kajin does not reduce Fehling solution as such but readily does so after hydrolysis with dilute mineral acids. The dried sample, on analysis, gave C, 58.7; H, 4.9; active H, 0.7;  $C_{19}H_{20}O_9$  requires: C, 58.1; H, 5.1; active H (for 3 H) 0.7; M.W., 392.  $C_{19}H_{18}O_9$  requires: C, 58.6; H, 4.6 per cent; M.W., 390.

*Acetyl-kajin*,  $C_{27}H_{26}O_{12}$  — 0.2 gm. of kajin with 3 c.c. of acetic anhydride and 0.6 gm. of fused sodium acetate were heated together on a water bath for 4 hr. The reaction mixture was diluted with cold water and kept aside for some time when the acetyl derivative precipitated out as a white, granular powder. It was filtered, washed with water and crystallized from methanol, when acetyl-kajin was obtained in the form of colourless needles melting at 122°C. (yield, 0.2 gm.).

Acetyl-kajin is readily soluble in ethyl acetate, acetone, alcohol and methanol, and insoluble in petroleum ether, ether and benzene. It does not give any colouration with ferric chloride and is insoluble in dilute alkali. After drying to constant weight at 61°C. *in vacuo* over phosphorus pentoxide, it gave C, 59.1; H, 5.0; M.W. (Rast), 545;  $C_{27}H_{26}O_{12}$  requires: C, 59.8; H, 4.8 per cent; M.W. 542.

#### Hydrolysis of Kajin with Baryta Solution

*Kajinin*,  $C_{15}H_{12}O_5$  — 0.1 gm. of kajin was heated on a water bath for an hour with a saturated solution of baryta. The reaction mixture was saturated with carbon dioxide and the precipitated barium carbonate was filtered and washed with water.

The combined filtrate and washings were acidified with dilute sulphuric acid and shaken out with ethyl acetate. The pale-yellow ethyl acetate layer was washed with water, dried over anhydrous sodium sulphate and freed of the solvent. The residue (0.06 gm.) was repeatedly crystallized from dilute alcohol, when it finally yielded the pure aglucone, kajinin, in the form of colourless needles melting at 208°C., decomp.

Kajinin dissolves in dilute sodium bicarbonate solution with effervescence in dilute alkali to a pale-yellow solution and gives a reddish-brown colouration with ferric chloride in alcoholic solution. In concentrated sulphuric acid, it dissolves to an orange solution, and in concentrated nitric acid with a greenish-yellow colouration. It does not contain any methoxy group and is optically inactive in alcoholic solution. After drying to constant weight *in vacuo* over phosphorus pentoxide at 100°C., it gave C, 63.1; H, 5.1;  $C_{15}H_{12}O_5$  requires: C, 62.9; H, 4.8; M.W., 248.  $C_{15}H_{10}O_5$  requires: C, 63.4; H, 4.0 per cent; M.W., 246.

*Acetyl Kajinin* — 0.05 gm. of kajinin, 1 c.c. of acetic anhydride and a few drops of pyridine were refluxed at 135°C. for 2 hr. After working the reaction product in the usual manner, the acetyl derivative was obtained from dilute alcohol in clusters of needles melting at 200°C. (yield, 0.04 gm.). It is soluble in methanol, alcohol, acetone and ether, and insoluble in petroleum ether, benzene and water.

*Methyl Kajinin* — A mixture of 0.1 gm. of kajinin in 7 c.c. of dry acetone, 0.5 gm. of anhydrous potassium carbonate and 4 c.c. of methyl iodide was refluxed at 40°C. for 4 hr. The reaction mixture was acidified with dilute hydrochloric acid, freed of most of the acetone on a water bath and then extracted with ethyl acetate. The ethyl acetate solution was washed with water, dried over anhydrous sodium sulphate and freed of the solvent. The residue, on repeated crystallizations from dilute alcohol, finally gave colourless needles melting at 178°C., and soluble in methanol, alcohol, acetone and ether, and insoluble in petroleum ether and water.

*Sugar Component* — The aqueous acidic layer, obtained from the baryta hydrolysis of kajin, was neutralized with barium carbonate and filtered. The filtrate, on concentration and subsequent heating with phenylhydrazine hydrochloride and sodium acetate in

aqueous solution, gave an osazone which melted at 202°C. and did not give any depression in the melting point on admixture with an authentic sample of glucosazone.

**Estimation of Glucose**—0.0691 gm. of kajin was hydrolysed with dilute sulphuric acid and the reaction mixture was boiled with Fehling solution. The precipitated cuprous oxide was filtered through a Gooch crucible, washed and dried. Found : cuprous oxide, 0.0269 gm., equivalent to 47.3 per cent glucose;  $C_{18}H_{20}O_6$  requires for 1 mol. of glucose, 45.9 per cent.

**Gallic Acid**,  $C_7H_6O_5 \cdot 2H_2O$ —The total aqueous alcoholic filtrate from crude kajin and the petroleum ether washings were taken together and extracted with petroleum ether. The dilute alcoholic layer was evaporated to dryness *in vacuo*. The reddish-brown semi-solid mass interspersed with crystals, which was left as residue, was exceedingly soluble in water, methanol and alcohol, and insoluble in ether, petroleum ether and ethyl acetate. It was digested with a small quantity of dilute hydrochloric acid and the acidic solution repeatedly extracted with ether. On removal of the solvent from the combined ethereal extracts, a crystalline residue was obtained which, on repeated crystallization from glacial acetic acid, finally yielded gallic acid melting at 241°C., decomp. (yield, 2.7 gm.); gallic acid, m.p., 241°C.

It dissolved in dilute bicarbonate solution with effervescence and gave an inky-blue colouration with ferric chloride. It reduced Fehling and ammoniacal silver nitrate solutions, gave a pink colouration with an aqueous solution of potassium cyanide, and a carmine red with ammoniacal potassium ferricyanide. After drying to constant weight *in vacuo* at 100°C. over phosphorus pentoxide, it lost 20.4 per cent in weight. The dried sample on analysis gave C, 49.5; H, 3.6;  $C_7H_6O_5$  requires : C, 49.4; H, 3.5 per cent; M.W., 170.

The identity of the acid with gallic acid was further confirmed through its decarboxylation to pyrogallol and the mixed melting points of its acetyl and methyl derivatives, methyl ester and semicarbazone with the corresponding derivatives of gallic acid.

**Kajidin**,  $C_{14}H_{14}O_8 \cdot H_2O$ —The cream-coloured, micro-crystalline powder obtained during the isolation of kajin, as described at the outset, was dissolved in a small quantity of alcohol and the solution was treated with

ether and petroleum ether to precipitate the coloured impurities. The clarified solution was freed of the solvent, first on the water bath and finally *in vacuo*. The granular powder thus obtained was macerated with a small quantity of ice-cold water to remove the water-soluble impurities and then desiccated *in vacuo* (yield, 26.0 gm. from 4700 gm. of the shells).

Kajidin, as obtained in the manner described above, is an optically inactive, cream-coloured, micro-crystalline powder, which melts at 185°C. with decomposition. It is soluble in methanol, alcohol, acetone and ethyl acetate, and insoluble in ether, petroleum ether, benzene and chloroform. It dissolves in ammonia or dilute alkali with a yellow colouration and is precipitated unchanged on acidification of the solution. It gives an inky-blue colouration with ferric chloride and readily reduces Fehling solution and ammoniacal silver nitrate. On drying to constant weight at 61°C. *in vacuo* over phosphorus pentoxide, it lost 6.5 per cent in weight. The dried substance gave C, 54.1; H, 5.0; active H, 1.6;  $C_{14}H_{14}O_8$  requires : C, 54.2; H, 4.5; active H (for 5 H), 1.6 per cent.

The acetyl derivative prepared in the usual manner on drying to constant weight *in vacuo* over phosphorus pentoxide at 100°C. gave C, 55.6; H, 4.7; M.W. (Rast), 481.  $C_{22}H_{22}O_{12}$  requires : C, 55.3; H, 4.6 per cent; M.W., 478.

On catalytic reduction with platinum black, it absorbed hydrogen equivalent to one double bond, but no crystalline product could be obtained from the solution.

**Hydrolysis with Baryta Solution**—1 gm. of kajidin was heated for an hour with saturated baryta solution on a water bath. The reaction mixture was acidified with dilute hydrochloric acid and extracted with ethyl acetate. The ethyl acetate solution gave a residue which, on crystallization, yielded gallic acid (yield, 0.5 gm.).

The aqueous acidic solution did not reduce Fehling solution and failed to give an osazone.

#### Anacardin

4 kg. of cashew shells from nuts stored for nearly 10 years were extracted with alcohol, and the total extract was worked up in a similar manner as in the case of kajidin. The fraction corresponding to kajidin was obtained in the form of a light brown, micro-crystalline powder melting at 195°C., decomp. (anacardin).

Its solubility, colour reactions with ferric chloride and its behaviour towards alkalis were similar to those of kajidin. After drying to constant weight at 100°C. *in vacuo* over phosphorus pentoxide, it gave C, 59.0; H, 5.2 per cent.

On reduction with platinum black, anacardin gave a colourless crystalline product melting at 88°C. in a yield of 25 per cent. The identity of this product with tetrahydro-anacardic acid was proved through its analysis and a comparison of the melting point and mixed melting points of the two products and their acetyl derivatives.

On hydrolysis with baryta solution, anacardin yielded gallic acid in a yield of only

8 per cent as against 50 per cent obtained in the case of kajidin.

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## Dry Cyclone Filters\*

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**A**MONG the various devices<sup>1</sup> employed for the filtration and separation of industrial dust from air, dry cyclones have received much attention. The use of cyclone collectors for collecting sand, sawdust, grain, sugar and similar other materials of sizes up to 100 $\mu$  is well known. "Aerotec" tubes in parallel have been employed by Yellott and Singh<sup>2</sup> in their work on gas turbines for the separation of ash from powdered coal at high temperatures. A cyclone separator<sup>3</sup> has been employed in these laboratories for controlling the particle size of carbon used in the manufacture of carbon electrodes for dry cells.

During the past twenty years or so, a considerable amount of work on the flow patterns and the mechanism of dust removal in cyclone collectors has been carried out<sup>4-11</sup>. The flow patterns of gas inside a cyclone are the double spiral and the double eddy. In the former, the flow pattern consists of an outer spiral moving downwards from the cyclone inlet and an inner spiral of smaller radius moving upwards into and through the exit pipe. The double eddy flow pattern

of gas inside a cyclone was noted and investigated by Van Tongern and has been reported by at least one other worker<sup>12</sup>. The theory of the latter type of flow has not yet been thoroughly worked out.

Dry cyclones are usually considered to be suitable for coarse dusts only, but when properly designed and applied, these units will give efficient and economic performance when handling sub-sieve dust particles as small as 10 $\mu$ . They combine the advantages of high collecting efficiency, simplicity, low pressure drop and low initial and operating costs. A specially noteworthy feature is their fool-proof working. It was, therefore, felt desirable to design and to construct cyclone filters suitable for handling particles up to 10 $\mu$  in these laboratories. They are described in this paper.

#### Experimental

In his article on "Centrifugal Dust Collectors"<sup>13</sup>, Larcombe suggested a design for high efficiency cyclones. His curves were extrapolated to suit our requirements and utilized in the design of two cyclone filters,

\* Paper read before the 36th Session of the Indian Science Congress, Allahabad (1949).



A and B. Two cyclones of group A suitable for 50 cu. ft. per minute air flow, and two of group B to handle 25 cu. ft. per minute were constructed. Cyclone A and its components are diagrammatically illustrated in Fig. 1. A reservoir of sufficient capacity for the collected dust is provided at the bottom of the cyclones so that it is able to function efficiently for 6 hr. or more at a stretch. To facilitate the easy removal of the accumulated dust, the sides of the reservoir are suitably tapered. A suitable arrangement

service. The weights of the two cyclone filters are 10 lb. and 6.5 lb. respectively.

Tests on the collecting efficiency, particle size retention capacity and pressure drop of both the filters were conducted. An electric blower was connected to the inlet duct of the cyclone. A dust sample comprising of particles of sizes ranging from 1 to  $1,000\mu$  was dusted at the air inlet of the blower. A large quantity of the dust collected in the filter. It was weighed to ascertain the collecting efficiency of the cyclone. The particles, which were not arrested by the cyclone, were collected on a filter-paper baffle soaked with lubricating oil and held at the outlet end. The filter paper was examined under a microscope to ascertain the maximum predominant particle size. Four tests at different rates of air flow (adjusted by varying the area of the air inlet of the electric blower) were carried out and the results obtained with cyclone A are summarized in Table I.

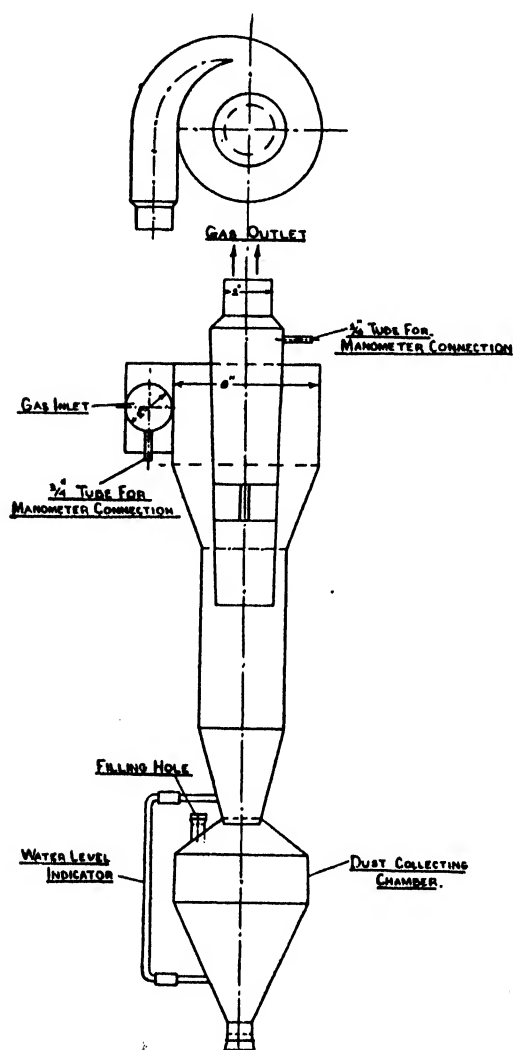


FIG. 1 — CYCLONE FILTER.

for connecting a manometer across the inlet and outlet ducts facilitates the measurement of pressure drop when the cyclone is in

TABLE I—SIZE OF DUST PARTICLES PASSING THROUGH CYCLONE FILTER 'A' AT VARIOUS RATES OF AIR FLOW

FLOW, cu. ft. per min.	MAXIMUM PREDOMINANT PARTICLE SIZE COLLECTED ON FILTER PAPER AT DIFFERENT POSITIONS, microns						MEAN DIAMETER OF PARTICLE, microns
	28	32	28	30	22	30	28
20	28	32	28	30	22	30	28
30	18	20	22	25	20	22	21
40	18	15	15	12	30	12	15
50	12	14	10	12	14	12	12

Error of measurement =  $2\mu$ .

The efficiency of a cyclone for one particle size is different from that for another at any given throughput of air. A convenient method for expressing cyclone efficiency is to plot a curve for particle size against efficiency at that size. Three samples of dust having maximum predominant particle sizes of 20, 69 and  $150\mu$  were prepared. The outlet pipe of the cyclone filter was connected to the inlet end of the conical filter unit<sup>14</sup> with a standard filter paper<sup>15</sup> clamped in between the two cones. The engine of the bench test unit was made to draw a constant flow of air at the rate of 50 cu. ft. per minute through the cyclone filter. A known weight of dust from each of these samples was dusted at a uniform rate at the inlet end of the cyclone filter. A major portion of it collected in the reservoir and was weighed. Dust which passed through, was collected on the filter paper, and was examined



under a microscope for maximum predominant particle size. The results of the various tests with filters A and B are summarized in Table II and graphically represented in Figs. 2, 3 and 4.

**TABLE II—COLLECTING EFFICIENCY & SIZE OF PARTICLES PASSING THROUGH CYCLONE FILTERS A & B**

PARTICLE SIZE OF DUST SAMPLES, MICRONS	COLLECTING EFFICIENCY, %	MAXIMUM PREDOMINANT SIZE OF PARTICLE ON FILTER PAPER, MICRONS	
			B
20	78	88	5
69	97	98	7
150	96	98	6

Error of measurement =  $2\mu$ .

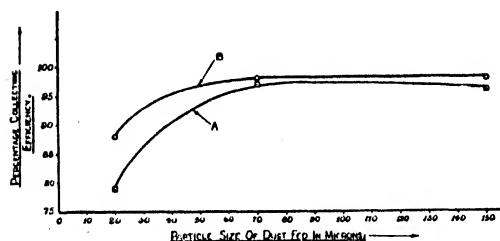


FIG. 2

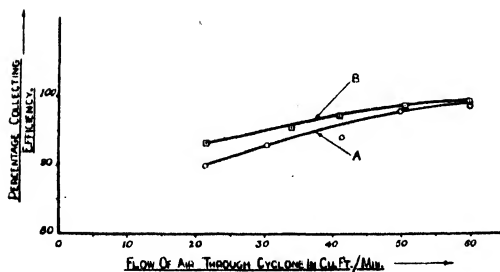


FIG. 3

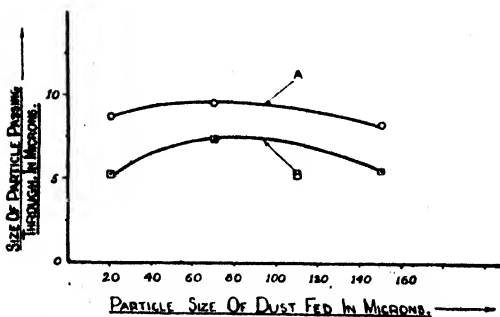


FIG. 4

Pressure drop for clean air at various rates of flow for both the cyclone filters are shown in Fig. 5.

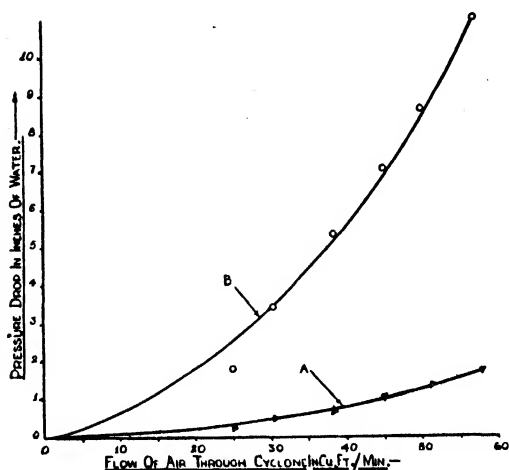


FIG. 5

## Discussion

In evaluating the performance of a cyclone, the collecting efficiency and pressure drop are the prime factors. When handling industrial dust, the collecting efficiency alone is the problem for the designer. In designing cyclone filters, both the factors have to be taken into consideration.

Overall efficiencies of 99 per cent have been recorded by various workers with particles above  $100\mu$ . A study of Table II shows that both cyclone filters A and B are capable of arresting dust particles of sizes up to 9 and  $6\mu$ , with collecting efficiencies of 97 and 98 per cent respectively, when dusts having particles of sizes up to  $150\mu$  are fed in. The pressure drop across the cyclone filters A and B is a few inches of water. The filters are very light to handle.

It is known that the pressure drop across a cyclone is less when dust is present in the air stream than when the air is clean. The pressure drop decreases and the collecting efficiency increases with increase in the dust concentration of the air stream. Under normal working conditions, these filters will give a better performance.

These filters have been used with advantage in portable producer-gas filtration systems.

An application for a patent covering the cyclone filters described in this paper has been filed.

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### Summary

Two cyclone filters, one for 50 cu. ft. per minute and another for 25 cu. ft. per minute air flow and capable of handling dusts of particle size up to  $10\mu$  with a collecting efficiency of 98 per cent, have been designed and constructed. Low pressure drop and light weight are the other features of these filters.

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## The Scope of Dry Cyclones in Portable Producer-Gas Plant Filters\*

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DETAILED studies of fibrous materials<sup>1</sup> and textile fabrics<sup>2</sup> for use in the various stages of gas filtration of a mobile gas producer, as adopted in India, have been previously described. The correct packing and proper use of these materials inside the filter cages to prevent channelling of gas requires much time and skill on the part of the plant operator. The need for frequent cleaning and renewal of the filtering media and their susceptibility to charring are but two of the serious drawbacks encountered in practice.

It is a normal practice to employ coir for the first, cotton waste in the second and satin drill cloth bag for the final stages of producer-gas filtration. In all these types of filters, the pressure drop that is required to pass a given quantity of gas through them is an important factor for consideration.

During the gas plant operation, the dust deposited in the filters continuously raises the pressure drop across the filtration system, and the volumetric efficiency of the engine decreases rapidly. Secondly, as the quality of charcoal generally employed varies, the rate of increase in pressure drop is unpredictable. For maximum efficiency of operation, it is desirable to have the least amount of pressure drop through the filtration system.

Of the various devices<sup>3</sup> employed for the filtration of industrial dust from air, dry cyclones and wet scrubbers appear to hold promise for producer-gas filtration. The pay load of the mobile vehicle is a major consideration in the selection of the filters and dry cyclone filters, in virtue of their comparative lightness, satisfy this requirement admirably.

\* Paper read before the Engineering and Metallurgical Section of the 36th Indian Science Congress Session, Allahabad, 1949.

The manufacturers of "Simpson" and "Victory" producer-gas plants have provided a cyclone filter in the initial stages of gas filtration along with the usual filters, probably to prolong the working hours of the gas plant. Preliminary tests with these filters showed that they are capable of removing the coarser dust particles with a collecting efficiency of about 70 per cent. A more efficient unit should obviously prove advantageous. In the previous communication<sup>4</sup>, two cyclone filters, one (A) for 50 cu. ft. per minute and the other (B) for 25 cu. ft. per minute air flow and to handle dust particles of sizes up to  $10\mu$ , have been described. To overcome the various difficulties experienced in the use of coarse and fine filtering media in the various stages of producer-gas filtration, the use of these filters suggested itself. These filters are fool-proof in their operation and are free from the many drawbacks of the filters commonly employed for producer-gas filtration.

### Experimental

Cyclone filters A and B, either alone or in combination, or in conjunction with the filters in vogue, were employed to replace the original cotton waste and cloth bag filters of a T.V.S. down-draught gas plant used in the various departmental trials<sup>5</sup>. The gas plant provided with the new filters was subjected to gas purity bench tests according to Indian Standard Specifications<sup>6</sup>. A large number of trials with various combinations of filters was undertaken to arrive at a simple, convenient and efficient combination suitable for a mobile gas producer. A few of these combinations together with the relevant details and the results of bench tests are summarized in Table I.

### Discussion

In evaluating the performance of a cyclone in a mobile gas producer, both the collecting efficiency and the pressure drop are of equal importance. As previously reported<sup>4</sup>, both cyclone filters A and B have shown an overall collecting efficiency of 98 per cent, and are light in weight. They are capable of arresting dust particles of sizes up to  $9\mu$ , while coir and cotton waste, when packed to a depth of 2" in a linear filter, are capable of arresting particles only up to  $281\mu$  and  $37\mu$ . It has been already stated that under the normal working conditions of a gas plant, filters A and B would show an increase in the

collecting efficiency and a decrease in the pressure drop.

The results of bench tests (TABLE I), using combination of dry cyclone filters A and B in place of the usual filters of a gas plant, show that the gas plant is not able to pass the prescribed gas purity dust tests. These filters, even in combination with other filters, as adopted in tests 11, 12 and 13, are unsatisfactory. Their failure was due either to a choking of the filters thereby causing an increase in the pressure drop across the producer, or to a high pressure drop across the filter paper in the conical filter unit. On the other hand, when water was used in the dust-collecting reservoir provided at the bottom of the cyclone, the dust retention capacity increased considerably (tests 8, 9 and 10). The collecting efficiency increased further when a wetting agent, such as "Permal WA", "Calsoline H.S. oil", etc., was used in small concentrations. This exceptional behaviour of the filter unit should be attributed to an increase in humidity inside the cyclone filter. Further, the tendency of dust particles to be carried away by the air stream, which has a tremendously high velocity inside the cyclone filter, is reduced to a minimum when the dust particles are wetted.

It is clear from the results given in Table I that dry cyclone filters containing water in the reservoir cannot pass the prescribed gas-purity dust test unless they are supplemented by cotton waste or cloth bag filters. As previously stated<sup>7</sup>, this is due to the fact that it is difficult to remove the last traces of fine dust in the gas, when using charcoal as the fuel, by the use of dry cyclone filters alone. Chowdhury<sup>8</sup>, while working with dry cyclone filters alone, was faced with the same difficulty. This observation is further supported by the work of Bowden and Kennedy<sup>9</sup> on wear in producer-gas converted petrol vehicle engines.

A further study of Table I shows that the combinations of filters, as adopted in tests 8, 9 and 10, are satisfactory. When employing the combination of filters as specified in test 8, the rate of increase in pressure drop across the producer during the gas-purity dust test was 0.1" of Hg per hour. This system of filters when used on a road vehicle is expected to give satisfactory performance for 80 hours as compared to the other two combinations of filters, which can run satisfactorily for 8 hours only, even for best setting. The time required to clean the

TABLE I—RESULTS OF GAS-PURITY BENCH TESTS ON CYCLONE FILTERS USED IN CONJUNCTION WITH OTHER FILTERS IN A T.V.S. DOWN-DRAUGHT GAS PLANT

TEST No.	ARRANGEMENT OF FILTERS	CALORIFIC VALUE AT N.T.P. PER CU. FT. B.Th.U.	DURATION OF DUST TEST, min.	DUST CONCENTRATION mg./cu. in.	PRESSURE DROP ACROSS PRODUCER AT THE END OF DUST TEST, in. of Hg.	PRESSURE DROP ACROSS FILTER PAPER AT THE END OF DUST TEST, in. of Hg.	REMARKS
1.	Cyclone filter A; water filter	89.5	5	155	...	23.4	Failed
2.	Cyclone filters A and B; water filter	116.2	5	102	...	>10	do
3.	Cyclone filter A; water filter; felt filter	110.0	20	43	...	>10	do
4(a)	Cyclone filters A and B; water filter; cloth bag filter	108.0	5	98	2.8	9.5	do
4(b)	Do	110.0	10	20.8	10.8	2.3	do
5.	Cyclone filters A, B, B; felt pad filter	...	10	24	3.2	9.2	do
6.	Cyclone filter A; water filter; cyclone B; felt pad filter	...	5	80	2.0	11.5	do
7(a)	Cyclone filters A, B, B; felt pad filter; cloth pad filter	...	15	3.2	10.8	1.4	do
7(b)	Do	...	5	2.7	13.8	1.7	do
8(a)	Cyclone filters A and B with water in reservoir, cotton waste in a radial filter	77	30	10	3.6	4.1	do
8(b)	Do	98.3	30	1.5	3.9	2.0	Passed
8(c)	Do	90.5	30	3.7	3.4	2.8	do
9.	Cyclone filter A with water in reservoir; felt filter; cloth bag filter	100	30	2.3	5.0	2.9	do
10(a)	Cyclone filter A with water in reservoir; cloth bag filter	105	30	3.3	7.2	7.5	Failed
10(b)	Do	118	30	2.2	5.1	2.85	Passed
11.	Cyclone filter A; cloth bag filter	...	5	...	3.2	10.5	Failed
12.	Cyclone filters A and B; cloth bag filter	112	30	24.8	4.8	2.8	do
13.	Cyclone filter A, jute in a radial filter; cloth bag filter	94.5	30	27.6	2.5	6.1	do

NOTE—The same number is used to designate a particular combination of filters, any minor alterations in the arrangement, or any modification in operation such, for instance, as the use of a wetting agent is indicated by adding a suffix (a), (b) or (c) to designate the number.

filtration system, adopted in test 8, is considerably reduced and one has not much to depend on the cleaner for the servicing of the filters. This combination of filters can be safely recommended for use with a portable producer-gas plant using charcoal as fuel.

In conclusion it may be stated that dry cyclone filters A and B in combination and containing water in the reservoir, with or without the use of wetting agent, can replace satisfactorily the coir and cloth bag or coir and cotton waste filters commonly used in the first and second stages of producer-gas filtration.

The cyclone filter described in this paper is covered by an Indian Patent.

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# Contact Angle of Natural Graphite

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THE study of contact angles of mineral surfaces in the three-phase system, air-water-solid, has played an important part in the development of froth flotation for the beneficiation of low grade ores. Contact angles of sulphide minerals have been measured by Wark<sup>1</sup> and Siedler<sup>2</sup>; they have been found to be zero (0) in distilled water, showing that the minerals are not inherently floatable. Eschenbach, Petersen and Popperle<sup>3</sup> have determined the contact angles of barytes, fluor spar, calcite, quartz and zinc blende in distilled water, and found that they vary from 50° to 70°. These specimens were presumably contaminated, and the values were accordingly unreliable<sup>1</sup>. Contact angles of coal have been measured by Brady and Gauger<sup>4</sup>, who found that the values vary from 20° to 54° depending upon the method of preparation of the coal surface. The study of contact angles of graphite appears to have attracted little attention.

During our work on the beneficiation of natural graphite, we were faced with the question whether graphite is inherently floatable or not. It has been reported by Gaudin<sup>5</sup> that graphite could be floated only in the presence of a frother and in the absence of a collector. Wark and Cox<sup>6</sup> have shown that the frothers commonly used in graphite flotation also have the properties of collectors. They have shown that the quantity of a frother, e.g. pine oil, necessary to float graphite in the absence of a recognized collector is much greater than when the collector is present. They have further shown that certain frothers like alcohol, acetone or cyclo-hexanol did not float graphite at all in neutral solution, although a good froth was formed.

It was, therefore, considered useful to measure the contact angle of graphite in the air-water-solid system.

The contact angle of mineral surfaces is measured by one of the following methods: (a) protractor measurements; and (b) spheroidal segment.

*Protractor Measurements* — This is employed in the captive-bubble apparatus developed by Taggart, Taylor and Ince<sup>7</sup>. It consists in adjusting the position of a gas bubble in the solid-liquid-gas system until it comes in contact with the solid surface submerged in the liquid. The contact angles can be measured by a protractor on an enlarged image of the bubble.

*Spheroidal Segment* — Mack<sup>8</sup> has shown that contact angles can be calculated from the dimensions of small bubbles adhering to a solid immersed under water.

The spheroidal segment method was adopted in our work, and an apparatus similar to that of Brady and Gauger<sup>4</sup>, with suitable modifications, was constructed.

*Apparatus* — A rectangular glass cell, 3×3×3 cm., held in a brass platform fitted to a stout stainless steel retort stand was employed. The platform could be adjusted to any height and kept in position by a tightening screw. A polished specimen of graphite, 2×2×2 cm., was suspended in the cell by 2 gold-plated copper wire bridges. A pyrex capillary tube, 1 mm. bore, was drawn into a thread with outside dia. c. 0.1 mm., and bore dia. 0.02 mm., and the drawn end was bent at right angles. The capillary tube was held in position by the mechanical stage of a low-power microscope used for magnifying the bubbles. The mechanical stage was held rigidly by a clamp fitted to the same retort stand. The capillary tube was held in such a way that the drawn end was dipped into the cell and was directly under the polished surface of the mineral specimen. The other end of the capillary tube was connected with a record syringe by a rubber pressure tubing. The record syringe was adapted to introduce air bubbles through the capillary tube and was operated by a screw fitted to the same stand. The bubbles which attached themselves to the lower surface of the specimen were illuminated by a suitable light source, and an oil-smeared ground glass plate, interposed between the cell and the illuminator to diffuse the light. The

bubbles were focussed by a low-power microscope, with a micrometer in the eye piece, and the dimensions of the bubbles measured.

Care was taken to prevent contamination of the test surface.

Lumps of graphite, free from flaws and cracks, were selected from high grade materials of low ash content and cut by a saw into a parallelepiped approximately  $2 \times 2 \times 2$  cm. The piece was rubbed in a sand paper to the proper shape, and one of the faces was polished under distilled water on a ground glass plate kept in a glass tray. The glass plate and the tray were previously washed with chromic acid to render them grease-free, and the piece was held by fingers covered by rubber surgical gloves, also rendered grease-free. Fine particles, if any, adhering to the polished surface were eliminated by rubbing against a grease-free, wet linen pad.

The polished specimen was transferred to the cleaned cell containing distilled water, and suspended with the polished surface downwards from the two bridges of gold-plated stout copper wire. The bent end of

the capillary tube, previously cleaned with chromic acid, was brought under the specimen and moved across the surface horizontally by operating the mechanical stage. Simultaneously, bubbles were released by manipulating the piston of the record syringe. The base of the cell was lightly tapped to accelerate the spreading of the bubbles. After about 10 min., the base and height of the bubbles adhering to the under-surface of the specimen were measured by a low-power microscope. The procedure was repeated with different specimens.

The bubble dimensions were calculated in millimeters and the contact angle calculated from the formula :

$$\tan \frac{\phi}{2} = \frac{2h}{b}$$

where  $\phi = 180 - \theta$ ,  $\theta$  being the contact angle measured through water;  $h$  = height of the bubble, and  $b$  = base of the bubble.

The results are shown in Table I.

The results with specimens of high grade natural graphite from Travancore and Ceylon show that the contact angle varies from  $17^\circ 49'$  to  $25^\circ 8'$ , the value increasing with

TABLE I

45 divisions in the ocular scale = 1 mm.

SPECIMEN	BUBBLE, No.	BASE, No. OF DIV.	HEIGHT, No. OF DIV.	CONTACT ANGLE	MEAN VALUE
Travancore graphite ( ash, 3.6% )	1	9	23.5	$21^\circ 40'$	$17^\circ 49'$
	2	12	24	$28^\circ 24'$	
	3	6	27	$12^\circ 40'$	
	4	5	25	$11^\circ 24'$	
	5	8	23	$19^\circ 44'$	
	6	5	20	$14^\circ 14'$	
	7	7	24	$16^\circ 36'$	
Travancore graphite ( ash, 3.0% )	1	8	23	$19^\circ 44'$	$21^\circ 56'$
	2	8	25	$18^\circ 10'$	
	3	9	23	$22^\circ 08'$	
	4	10	22	$25^\circ 36'$	
	5	7	15	$26^\circ 18'$	
	6	7	22	$18^\circ 24'$	
	7	6	15	$22^\circ 36'$	
	8	6	15	$22^\circ 36'$	
Travancore graphite ( ash, 2.94% )	1	5	10	$28^\circ 40'$	$21^\circ 44'$
	2	7	17	$23^\circ 16'$	
	3	4.5	12	$21^\circ 14'$	
	4	3.5	9	$22^\circ 00'$	
	5	5	16	$17^\circ 44'$	
	6	6	15	$22^\circ 36'$	
	7	5	17	$16^\circ 42'$	
Ceylon graphite ( ash, 1.35% )	1	8	15	$33^\circ 00'$	$25^\circ 08'$
	2	6	13	$26^\circ 00'$	
	3	6.5	14	$26^\circ 08'$	
	4	4	9	$25^\circ 02'$	
	5	5	11	$25^\circ 36'$	
	6	3.5	11	$17^\circ 10'$	
	7	5	11	$25^\circ 36'$	
	8	8	20	$22^\circ 36'$	
Ceylon graphite ( ash, 0.87% )	1	8	28	$17^\circ 28'$	$25^\circ 28'$
	2	10	23	$24^\circ 30'$	
	3	13	21	$46^\circ 28'$	
	4	10	23	$24^\circ 30'$	
	5	13	25	$29^\circ 06'$	
	6	8	23	$19^\circ 44'$	
	7	7	24	$16^\circ 36'$	

the purity of the specimen. This leads to the conclusion that graphite is inherently floatable.

The results recorded here compare favourably with those obtained by Ghosh and Banerjee<sup>9</sup> who employed protractor measurements. The contact angle for Ceylon graphite (ash, 0.3 per cent) was found to be 23°.

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# Letters to the Editor

## TRITERPENIC COMPOUNDS ISOLATED FROM *EUPHORBIA* LATICES

NEWBOLD AND SPRING<sup>1</sup> ISOLATED FROM the resin of *Euphorbia resinifera* two isomeric alcohols, euphol and euphorbol, having the molecular formula  $C_{30}H_{50}O$  and melting at 116°C. and 126°-127°C. respectively. They considered euphol to be tetracyclic diethenoid, and Jeger and Krusi<sup>2</sup> subsequently showed that a close relationship exists between euphol and the elemi-acids. Dupont and Marc Julia<sup>3</sup> isolated from *Euphorbia balsamifera* yet another triterpenic alcohol, m.p. 175°C., from which they obtained a ketone  $C_{30}H_{48}O$ , m.p. 186°C., through oxidation with chromic acid. In a recent publication, Macdonald, Warren and Williams<sup>4</sup> have reported the isolation of euphol from *Euphorbia tirucalli*, *E. triangularis* Desf., and *E. ingens* E. Mey. They have further found that this alcohol contains an isopropylidene group not attached to a ring, and yields 1:2:8 trimethyl phenanthrene on selenium dehydrogenation. These results, they conclude, would place euphol in the elemi-acid group of triterpenes. With the publication of the paper by Macdonald *et. al.*, it would be of interest to present a review in this note of the work done in this laboratory on the constituents of *Euphorbia tirucalli*.

Working with samples of the *dried* latex of *Euphorbia tirucalli* received from South India and stored for some months, Kari-mullah and Dutta<sup>5</sup> isolated a new crystalline ketone melting at 119°-120°C. which was subsequently assigned the formula  $C_{30}H_{48}O$  and named as euphoron by Karimullah, Gopalachari and Siddiqui<sup>6,7</sup>. This ketone, which is the only ketonic substance so far isolated from *Euphorbia* latices, gave, on reduction with sodium and moist ether, two isomeric alcohols having the molecular formula  $C_{30}H_{50}O$  and melting at 108°-109°C. and 117°-118°C. respectively. These two alcohols, in view of their being derived from euphoron, were named as euphorol and *iso*-euphorol. Euphorol was the principal reduction product of the ketone, while *iso*-euphorol was obtained in a poor yield and could not be studied in detail. Subsequent investigations<sup>8</sup> with samples of fresh latex led to the isolation of *iso*-euphorol as the principal constituent in place of the ketone, which was obtained from dried, stored latex and could not be isolated from the fresh material. A comparison of *iso*-euphorol and derivatives prepared from it with euphol of Newbold and Spring and its corresponding derivatives show that euphol and *iso*-euphorol are identical.

Euphoron, in keeping with its ketonic nature, gives an oxime, m.p. 194°-195°C., a hydrazone, m.p. 149°-150°C., and a semi-carbazone, m.p. 178°-179°C. From its iodine







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